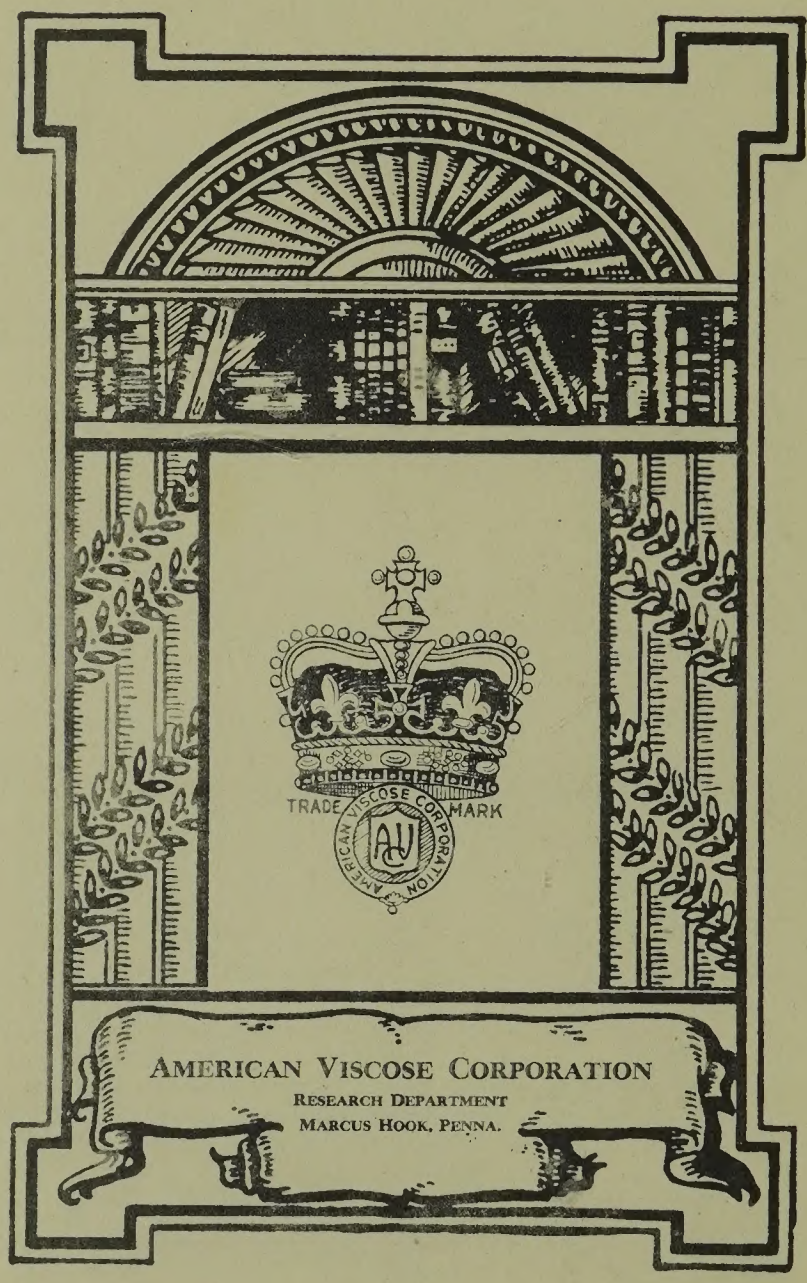
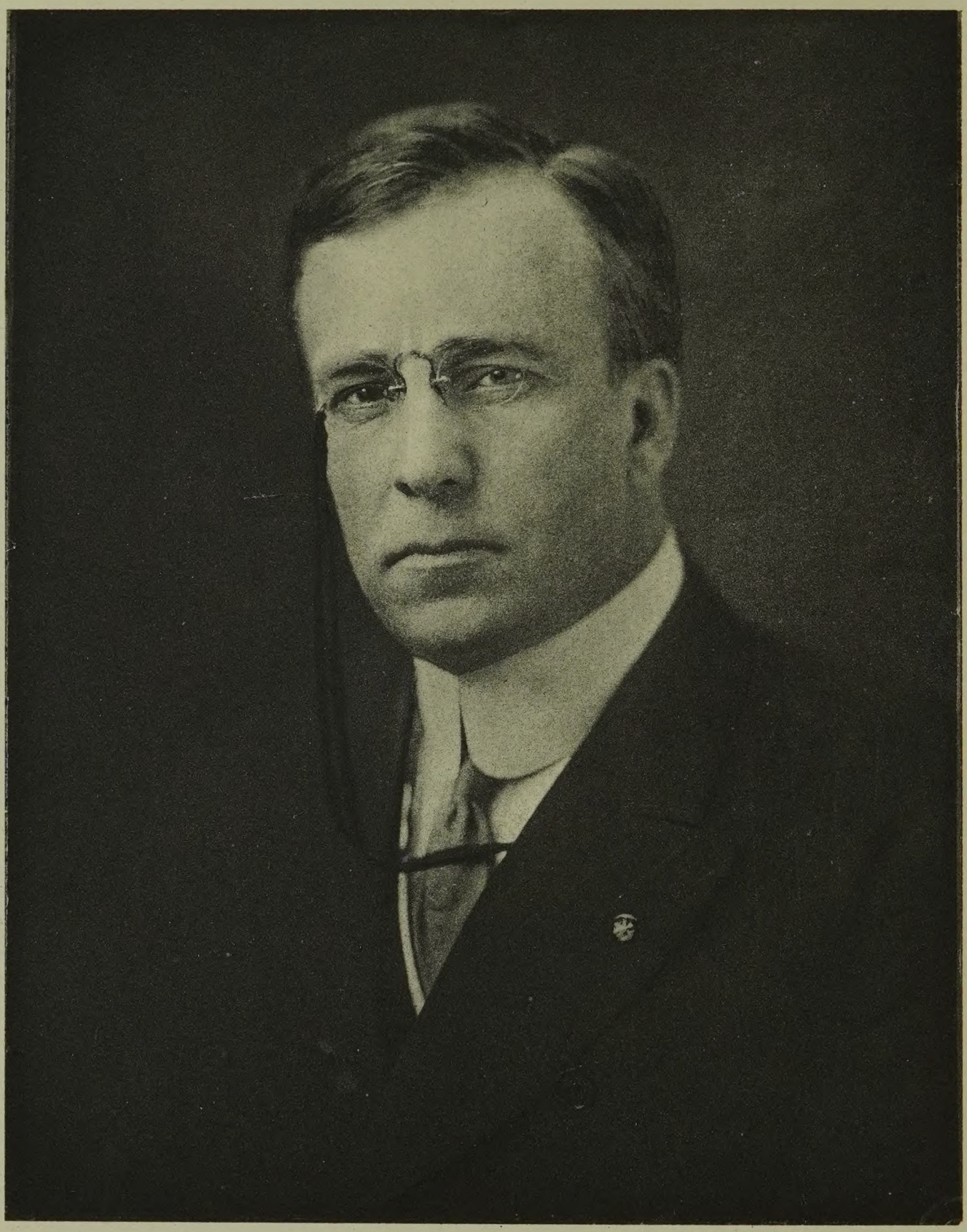
Edward Barton



PERIODICAL

The D. Van Nostrand Company intend this book to be sold to the Public at the advertised price, and supply it to the Trade on terms which will not allow of reduction.



T. R. Wagner.

TP
1
.C44X

(S) DM 1-16-81
Edward Barlow

TRANSACTIONS

OF THE

AMERICAN INSTITUTE

OF

CHEMICAL ENGINEERS

VOLUME V

1912

OFFICE OF THE SECRETARY
POLYTECHNIC INSTITUTE
BROOKLYN, N. Y.

PUBLISHED BY THE INSTITUTE
AND FOR SALE BY
D. VAN NOSTRAND COMPANY
NEW YORK
1913

The Institute as a body does not hold itself responsible for the statement of facts or opinions advanced in papers or discussions.

Copyright, 1913, by
AMERICAN INSTITUTE OF
CHEMICAL ENGINEERS

CONTENTS

ADDRESSES AND PAPERS READ BEFORE THE INSTITUTE

		PAGE
Phenol-Formaldehyde Condensation Products	L. H. BAEKELAND,	1
Protection of Intellectual Property in Relation to Chemical Industry	L. H. BAEKELAND,	19
Notes on a Study of the Temperature Gradients of Setting Portland Cement	ALLERTON S. CUSHMAN,	43
The Production of Available Potash from the Natural Silicates	ALLERTON W. CUSHMAN and GEORGE W. COGGESHALL,	52
Potash, Silica and Alumina from Feldspar	EDWARD HART,	68
A Chemical Investigation of Asiatic Rice	ALLERTON S. CUSHMAN and H. C. FULLER,	70
The Beehive Coke Oven Industry of the United States	A. W. BELDEN,	78
Action of Disinfectants on Sugar Solutions	GEORGE P. MEADE,	88
The Decomposition of Linseed Oil During Drying	J. C. OLSEN and A. E. RATNER,	100
Tests on the Opacity and Hiding Power of Pigments	G. W. THOMPSON,	108
Control of Initial Setting Time of Portland Cement	E. E. WARE,	119
The Effect of "Lime-Sulphur" Spray Manufacture on the Eyesight	JAMES R. WITHROW,	127
Acetylene Solvents	J. H. JAMES,	133
The New Chemical Engineering Course and Laboratories at Columbia University	M. C. WHITAKER,	150
The Need of Standard Specifications in Oils for Paving Block Impregnation	JOHN HAYES CAMPBELL,	170
The Presence of Oxygen in Petroleums and Asphalts	SAM. P. SADTLER,	178
The Chemical Engineer and Industrial Efficiency	WM. M. BOOTH,	184
Water for Industrial Purposes	WM. M. BOOTH,	197
The Availability of Blast Furnace Slag as a Material for Building Brick	ALBERT E. WHITE,	204

PAGE

Technical Accounting and Chemical Control in Sugar Manufacture. . . . . DAVID L. DAVOLL, Jr., 220
The Bituminous Rocks of the United States and Their Use for Street Surfaces. . . . . S. F. PECKHAM, 245
Code of Ethics. 255
Constitution. 259
Officers and Committees for 1913. 266
Membership List. 269

INDEX. 281

TRANSACTIONS OF THE AMERICAN INSTITUTE OF CHEMICAL ENGINEERS

PHENOL-FORMALDEHYDE CONDENSATION PRODUCTS

By L. H. BAEKELAND, ScD., Yonkers, N. Y.

Read at Joint Meeting with the Eighth International Congress, New York City, September 4-13, 1912.

The resinous or amorphous products resulting from the action of phenolic bodies upon formaldehyde have lately attracted considerable attention on account of their rapidly increasing applications for industrial purposes.[1]

It is questionable whether this general designation of "condensation products of phenols and formaldehyde" should be maintained much longer. Indeed, it is well known that these products can be obtained without the use of so-called formaldehyde. In fact, the first condensation products thus described were produced without the use of formaldehyde,[2] and it is generally accepted that other methylene compounds, for instance, methylal, trioxymethylen, hexamethylentetramin, etc., can replace formaldehyde in this reaction. The fact that hexamethylentetramin can suitably replace formaldehyde in the formation of the infusible phenolic condensation

[1] Baekeland: "The Synthesis, Constitution and Uses of Bakelite," Journal of Industrial and Engineering Chemistry, Vol. 1, No. 3, 1909, page 149. "On Soluble, Fusible, Resinous Condensation Products of Phenols and Formaldehyde," Journal of Industrial and Engineering Chemistry, Vol. 1, No. 8, 1909, page 545. "Recent Developments in Bakelite," Journal of Industrial and Engineering Chemistry," Vol. 3, No. 12, 1911, page 932.

[2] Berichte, 5, p. 1905; 19, pp. 3004 and 2009; 25, p. 2411.

products was published as far back as December 31, 1907, by Lebach.[3]

Lately, I have succeeded in producing fusible resinous condensation products identical with those described by Blumer, DeLaire, etc.,[4] by introducing a mixture of salicylic acid and an inorganic acid in the cathode compartment of an electrolytic cell in which sodium chloride is electrolyzed, a mercury cathode being used. According to the well know reaction of Kolbe, the carboxyl group of salicylic acid is introduced by reacting with CO_2 on phenolate of sodium. So that we have here an example of the possibility of introducing indirectly the methylene group as CO_2, then reducing the carboxyl group by means of nascent hydrogen. A similar observation has already been recorded by Velden,[5] who expected to get oxybenzyl-alcohol by reducing salicylic acid but obtained the corresponding saliretin-body resulting from the dehydration of phenol alcohol.

However, the designation "phenol-formaldehyde condensation products" has been so generally used, that for awhile at least, we shall have to submit to it as a matter of routine.

In the same way, we are erroneously designating as "formaldehyde" a commercial aqueous solution containing some real formaldehyde or methylen-oxide, CH_2O, with much methylenglycol, methylal, trioxymethylene, hydrates of trioxymethylene, other polyoxymethylens, etc., all compounds of methylen of which the technical value is equivalent in this reaction to that of true formaldehyde.[6]

The direct relationship of the resinous condensation products to phenol-alcohols or their anhydrides, seems now pretty well established. The so-called fusible soluble resinous condensation products are merely varieties of the saliretins,[7] and all these products differ

[3] Knoll patent, Belgium, No. 204,811, December 31, 1907. Ditto, Wetter (Knoll) British patent No. 28009, 1907, owned by the Bakelite Gesellschaft of Berlin.

[4] Baekeland, "On Soluble, Fusible, Resinous Condensation Products of Phenols and Formaldehyde."

[5] Velden, Journ. Prak Chemie., (2) 15, p. 164. Jahresbericht, 1877, p. 337.

[6] Raikov. Chem. Ztg., 26, 135; 12, 11 (1901). Kekule, Ber., 25, 2435. Harries, Ber., 34, 635. Compt. rend., 124, 1454; Bull. soc. chim. 17, 849. F. Auerbach, also Auerbach and Barschall, Arb. kais. Gesundh., Band XXII, Heft 3 and Band XXVII, Heft 1. Verlag Julius Springer, Berlin.

[7] Beilstein, Organ. Chemie, Vol. 2, 1896, p. 1109. R. Piria, Ann. Chem.,

from each other only by greater or lesser fusibility, solubility, or hardness, and each of these properties can be modified at will. Furthermore, we have the means at hand of producing all these bodies directly from phenol-alcohols.[8]

The formation of ortho- and para-oxybenzyl-alcohol, or their homologs, by Manasse and Lederer, is sufficiently well known.[9] This process consists in the direct action of one molecule of phenol on one molecule of formaldehyde in presence of one molecule of NaOH under special conditions.

Then DeLaire[10] showed that these same phenol-alcohols can be transformed industrially, by dehydration, into fusible resins or saliretin products suitable for commercial purposes in place of shellac, copal, or other natural resins. In that process, it is not necessary to first produce the phenol-alcohol in pure form, and the two reactions can be carried out practically at the same time, so that the phenol-alcohol is dehydrated to saliretin resins as soon as it forms.

This is accomplished more directly by reacting with phenol on formaldehyde in presence of an acid,[11] provided the reaction be carried out under suitable conditions. One of the required conditions is that the phenol should be in excess so as to avoid the formation of variable amounts of infusible and insoluble products. A fusible soluble saliretin can thus easily be prepared which has all the appearance of a resin; it melts if heated and solidifies by cooling; it is soluble in alcohol and acetone; it can be maintained in fusible condition for very long periods, without becoming infusible or insoluble, provided heating be carried on below certain tempera-

48, 75; 56, 37; 81, 245; 96, 357. Moitessier, Jahresbericht, 1886, p. 676. K. Kraut, Ann. Chem., 156, 123; Gerhardt, Ann. Chim. Phys. (3) 7, p. 215. F. Beilstein and F. Seelheim, Ann Chem. 117, p. 83. C. Schotten, Berichte, 1878, p. 784.

[8] Baekeland, "On Soluble, Fusible, Resinous Condensation Products of Phenols and Formaldehyde," Journal Industrial and Engineering Chemistry, Vol. 1, No. 8, 1909, p. 545.

[9] Journal Praktische Chemie (2), vol. 50, p. 224. Ber., 1894, 2409-2411; D. R. P., Bayer, 85588; U S. P., Manasse, 526,786, 1894.

[10] DeLaire, British Patent, 15517, 1905; D. R. P., 189,262.

[11] Blumer, Brit. Pat., 6823, 1903; 12880, 1902; DeLaire, French Patent, 361,539; Wetter (Knoll), Brit. Pat., 28009, 1907; Knoll, French Pat., 395,657; Bayer, D. R. P., 237,786; D. R. P., 201,261; Brit. Pat., 26317, 1907, etc.

tures, and provided the excess of phenol be not removed. This fusible resin and its preparation has been described by Blumer, and DeLaire as a "shellac substitute," or "resin substitute";[12] by Baekeland, who calls it "Novolak,"[13] and lately again by Aylsworth, who calls it "phenol resin."[14] In whatever way it be obtained, whether by using a phenol-alcohol (DeLaire, Baekeland); whether by starting from phenol and formaldehyde in the presence of oxyacids (Blumer), or in the presence of mineral acids (DeLaire, Thurlow, Bayer), or by the action of phenol on formaldehyde without adding condensing agents, (Story)[15] (or Aylsworth)[16] the product is absolutely the same in its chemical and physical properties. Its melting point or fusibility may be modified at will by varying the amount of free phenolic body. This free phenol exists in solid solution in the mass and can be eliminated by merely physical methods; by the partial elimination of this free phenol, the fusibility and the solubility of the resin are decreased. The last traces of free phenol cling tenaciously to these saliretin resins; so much, indeed, that at one time, I was inclined to believe that this small amount of phenol was chemically combined. Indeed, the last traces of phenol cannot be expelled by heating at the lower melting temperatures of the product. There is nothing strange in this, if we take into consideration that phenol itself has a relatively high boiling point, and we know of numerous examples where colloids retain, physically, small amounts of other bodies which form therewith colloidal solid solutions. As long as there is some excess of phenol present in the saliretin, it is possible to maintain the mass in fusion for a practically indefinite time, provided the temperature be not raised too high. Pure saliretin, containing no excess of phenol, may be kept in fusion for some time, but after awhile it polymerizes and becomes less fusible until finally it changes into some infusible product. However, it should be noted right here that this latter product, although it is infusible, does not possess the maximum mechanical strength nor hardness, nor general chemical and physical resistivity, of those other polymerized infusible products, of which I will speak

[12] Loc. cit.
[13] Journal Industrial and Engineering Chemistry, 1909, p. 545.
[14] U. S. Pat. 1,029,737.
[15] Austrian Pat. 30844, p. 2, l. 17 to 20.
[16] Loc. cit.

later on, and which are obtained by reacting with a sufficiently larger amount of formaldehyde, or equivalent methylen compounds, and which have been designated as Bakelite C.

It is possible to expel the slight excess of phenol which lends special fusibility to the so-called fusible resins; this can be accomplished whether these resins be called "shellac substitutes," "Novolak," or "phenol resins," or whether they be made directly from phenol alcohols, or from phenol and formaldehyde with or without acid condensing agents. Indeed, plain heating at 300° C. to 350° C. or better, heating in vacuo, or in a current of an inert gas, like nitrogen, easily expels the free phenol, and produces infusibility. The same result is readily obtained by entraining the free phenol, by blowing superheated steam through the molten resinous mass.

These facts are corroborating proofs to others which establish clearly the saliretin nature of these fusible products, and demonstrate their relationship to the phenol-alcohols from which they are derived. If these fusible resins are prepared directly from phenol and formaldehyde, the preliminary formation of phenol-alcohol may escape our notice, because by the action of heat on the mixture, especially in presence of acid bodies, the phenol-alcohols are rapidly dehydrated to saliretin products. But it is quite possible to demonstrate their presence, and my assistant, Dr. A. H. Gotthelf, while preparing fusible resinous condensation products, by boiling a mixture of phenol and formaldehyde, acidulated by means of HCl, has been able to extract from this mixture well defined crystals of oxybenzyl-alcohol before the heat had accomplished its resinifying action.

It is self-evident that as soon as high temperatures are applied to such mixtures, the formed phenol-alcohol will quickly undergo resinification, because the phenol-alcohol will be dehydrated to a saliretin product as soon as it is formed.

If there is an excess of phenol present, or if the formaldehyde reacts in insufficient proportions, which amounts to the same thing, a fusible saliretin will be the result.

In the absence of an excess of phenol, but using however, a restricted amount of formaldehyde or other methylen compound, a polymerized infusible saliretin will be produced.

But whenever we succeed in bringing into reaction a sufficient amount of formaldehyde or its equivalent, then a much harder,

much stronger and more resistive infusible body than an infusible saliretin will be formed, and this body of maximum strength and resistivity is identical with Bakelite C.

If we first produce the pure crystalline phenol-alcohol, containing no excess of phenol, for instance crystalline saligenin or oxybenzyl-alcohol, and if we heat it gently, it will simply dehydrate and be transformed in a fusible mass which on cooling, solidifies to a resinous product—a fusible saliretin resin. The latter, submitted to the further action of heat, polymerizes and becomes an infusible, insoluble saliretin. This polymerization is facilitated, by the presence of small amounts of catalyzers, for instance, hydrochloric acid. The presence of an excess of phenol retards polymerization; hence the infusibility induced by polymerization will be retarded, and this, until some way or another the excess of phenol has been expelled. The infusible polymerized saliretin obtained by heating phenol-alcohols containing no free phenol, or by heating fusible saliretin containing no free phenol, is insoluble in alcohol, but swells in acetone; it softens decidedly on heating, although it is no longer fusible. Longer heating does not harden it further, nor make it more resistive. It is harder, stronger, and more resistant to physical and chemical agents than the fusible saliretin from which it is derived; in this respect, it surpasses even more the soluble fusible resins described by Blumer,[17] DeLaire,[17] Baekeland,[17] and called "phenol resin by Aylesworth.[17] But even after polymerization or hardening has been carried as far as possible, it is considerably less hard and less strong and less resistant to physical and chemical agents than the polymerization products resulting from the reaction of phenol on a sufficiently large proportion of formaldehyde or equivalent substances.

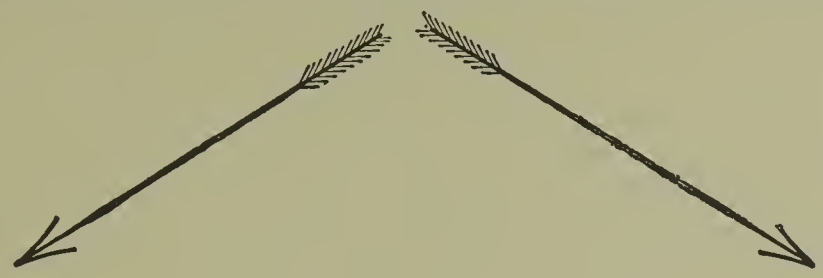
In order to obtain the latter polymerization products of maximum strength, hardness, and maximum resistivity, an adequately larger amount of methylen group must be introduced before or during the act of polymerization. The introduction of this methylen group may be accomplished by at least three distinct methods:

First method: Reacting directly with a sufficient amount of formaldehyde, or its equivalents upon phenol.

Second method: Reacting with formaldehyde or its equivalent on phenol-alcohol.

[17] Loc. cit.

PHENOL+FORMALDEHYDE (OR EQUIVALENTS) UNDER VARYING REACTING CONDITIONS FORM DIFFERENT BODIES.

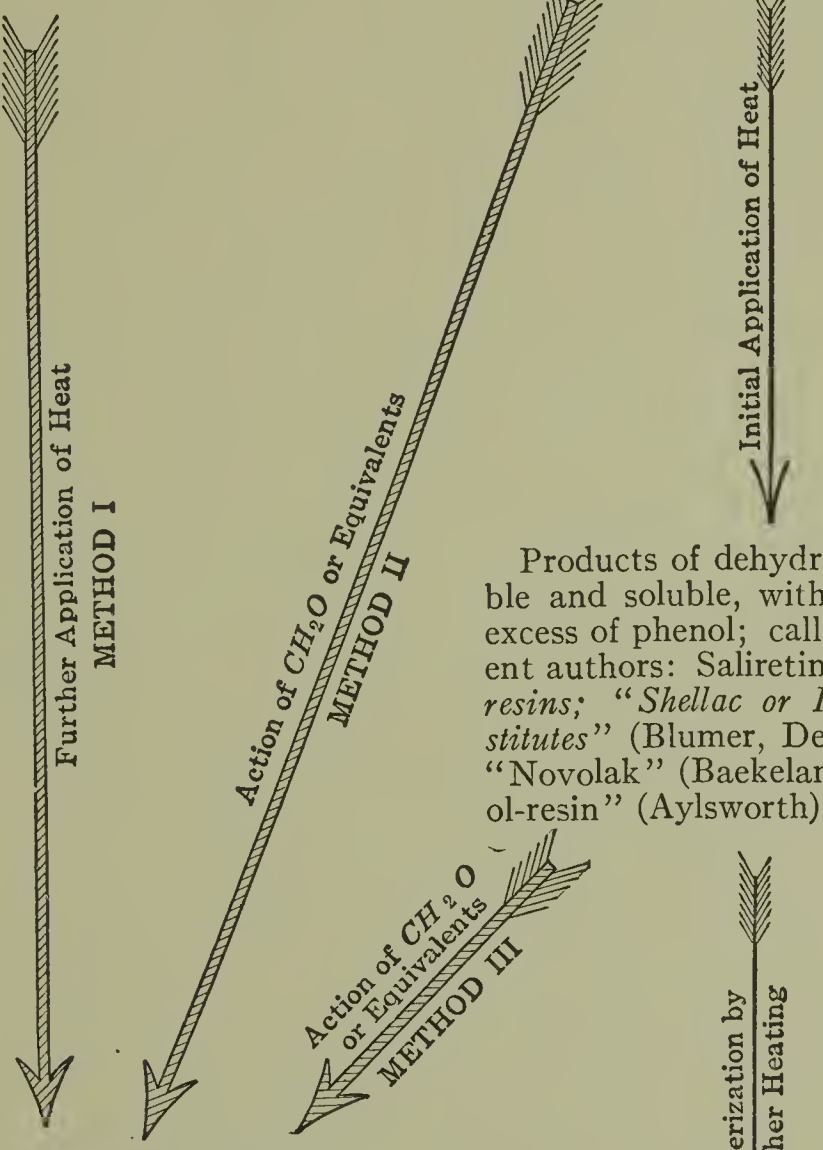


GROUP I

Initial or partial condensation productor Product A, liquid, pasty, or solid, but fusible and soluble.

GROUP II

Phenol-alcohols: oxybenzylalcohol saligenin, etc.



Product B or intermediate product: (Brittle when cold, elastic when hot, but infusible; swells in acetone without dissolving. By further application of heat is transformed in final Product C.)

Products of dehydration: Fusible and soluble, with or without excess of phenol; called by different authors: Saliretin, or *saliretin resins;* "*Shellac or Resin substistitutes*" (Blumer, DeLaire, etc.); "Novolak" (Baekeland); "Phenol-resin" (Aylsworth).

Final Product C, infusible, insoluble, and of maximum hardness, strength and resistiv ty. Called by different authors: "Bakelite C," "Final condensation product,' "Ultimate infusible product," "Inusible phenolic-condensation product." Hardness, strength, and resistivity decidedly superior to that of the end product of Group II.

Polymerized saliretins: substances of limited solubility or insoluble and of high melting-point or infusible. Less hard, less strong, and less resistive than Bakelite C.

Third method: Producing a fusible dehydration product of phenol-alcohols or saliretin products, then reacting thereon with formaldehyde or its equivalents.

The following diagram describes very well all these relations:

First or Direct Method. Direct Action of Phenol on Formaldehyde.

Phenol can be made to react on formaldehyde or its equivalents at sufficiently high temperature to produce directly an infusible insoluble polymerization product of maximum strength and hardness (Bakelite C). In this reaction, the main requirement is that a sufficient amount of formaldehyde or its equivalents should enter into reaction. For this purpose, it is not necessary nor sufficient that the required amount of formaldehyde be merely present, because some of the formaldehyde may not enter into reaction or be lost during the operation. The principal requirement is that the formaldehyde should react in sufficient amount on at least a portion of the phenol present, even if some of the latter remains uncombined in the mass.

By the use of suitable methods, this reaction can be interrupted at its initial stages, so as to produce initial or partial condensation products, which are temporarily fusible and soluble before further application of heat has changed them. These initial condensation products may be liquid, or pasty, or under specially favorable conditions, for instance, by the use of small amounts of some bases, they may be prepared in solid form. At any rate, they are soluble in alcohol and acetone, and the solid variety is fusible. These fusible soluble initial products should not be confounded with the further advanced and intermediate product B, as described in my paper on "The Synthesis, Constitution and Uses of Bakelite,"[18] because the latter is insoluble and infusible, although it has not acquired the maximum hardness and resistivity which further application of heat will bring forth by changing it to condition C.

Further action of heat upon these fusible and soluble initial condensation products will ultimately cause polymerization and produce the final infusible and insoluble product of maximum hard-

[18] Journal Industrial and Engineering Chemistry, Vol. 1, No. 3, 1909, p. 149.

ness, maximum strength, and maximum chemical resistivity. (Bakelite C).

By heating mixtures of phenol and formaldehyde in suitable proportions, at sufficiently high temperatures, for a sufficiently long time, chemical condensation may take place without the addition of condensing agents or catalytic agents; however, under such unfavorable conditions, the action is too slow and too difficult to control, for technical purposes.

By the addition of acids or acid salts, the reaction may be hastened to the point of becoming violent. But the presence of acid bodies tends to develop disturbing side-produce which lessen the technical value of the final product. Furthermore, in presence of the acid-reacting mixture, whenever there is sufficient excess of phenol, we do not obtain the infusible, final product, but resins of the fusible saliretin or "shellac substitute" type. Things go quite differently if small amounts of bases are present during the reaction.[19] Small quantities of bases prevent radically the formation of fusible soluble saliretin products (shellac substitutes, Novolak, phenol-resin, etc.) and insure the formation of infusible, insoluble, final products, even in presence of a decided excess of phenol. If the phenol be used in excess, it will be found in the final product as a solid solution. In fact, the excess of phenol may be exaggerated to the point that the final product becomes very flexible or assumes a gelatinous appearance, and swells considerably in certain solvents, like phenol, or alcohol, or acetone, without, however, entering into complete solution. In any case this free-phenol-containing substance is infusible; in other terms, the application of heat can no longer liquefy it, although higher temperatures may char or destroy it.

This behavior of the bases constitutes a radical difference with that of acids or other acid-reacting bodies. Whenever acid-reacting bodies are used in conjunction with an excess of phenol, or an insufficient amount of reacting formaldehyde (which amounts to the same thing), they tend to produce fusible, soluble resins, while under exactly the same conditions and with the same proportions of phenol and formaldehyde, small amounts of bases develop surely infusible polymerized bodies as ultimate products.

In this method, the bases should be used in relatively moderate

[19] See Baekeland, U. S. P., 942,809.

amounts; not in molecular proportions, as Manasse and Lederer[20] utilize them for making phenol-alcohols, nor as DeLaire[20] employs them to produce the fusible resinous dehydration products of these phenol-alcohols. Neither should they be used in such large quantities nor under such conditions as Hentschke[21] recommended for the manufacture of certain antiseptic compounds.

If the amount of base be properly restricted, the reaction proceeds very regularly and remains under easy control. The base acts as an excellent accelerator, both in the condensation and in the polymerization. The use of bases under above mentioned conditions enables one to carry out the reaction with utmost uniformity and certainty of results.

At first sight, the importance of these seemingly modest facts is not very apparent; no more than the value of the observation that sufficient counterpressure permits quick polymerization at high temperatures without risk of porosity; no more indeed than the realization of the fact that the final product with its great hardness, its strength and other splendid qualities, is unelastic and woefully deficient for most industrial purposes where great resistance to shock or vibration is required, and that the incorporation of suitable fibrous materials improves all this by modifying the shattering wave induced by impact. Yet these are the three main factors which have enabled us to harness into technical service an elusive laboratory reaction, and have rendered possible the creation of a new industry which is gaining daily in importance.

It has been shown[22] that small amounts of ammonia, or amines may be used to good advantage for this purpose. It is a well-known fact[23] that ammonia or ammonium salts, in presence of formaldehyde produces instantly a corresponding amount of hexamethylentetramin. In the same way, if any ammonia be added to a mixture of phenol and formaldehyde, a corresponding amount of hexamethylentetramin is produced which can easily be extracted from the mixture. This fact was confirmed by Lebach, and can easily be

[20] Loc. cit.

[21] Hentschke, D. R. P., 157,553.

[22] Baekeland, U. S. Pat., 942,809.

[23] Wohl, Ber., 19, 1892; Tollens, Ber., 17, 653; Carl Goldschmidt, p. 29; Bonn. Verlag von Friedrich Cohn, 1903; Cambier, Brochet, Compt. rend., 120, p. 557.

ascertained by direct experiment.[24] It is self-evident that instead of a mixture of phenol and formaldehyde and ammonia, an equivalent amount of hexamethylentetramin or hexamethylentetramin-triphenol[25] may be used. So that in the preparation of these bodies, formaldehyde can be replaced by hexamethylentetramin. This was already published by Lebach in the patent literature as far back as the end of 1907.[26] Whether the phenol mixture be prepared with ammonia or with hexamethylentetramin, its properties are practically the same, and on heating both mixtures engender the same product, with final evolution of ammonia gas.

Whatever be the methods employed, this reaction is strongly exothermic, and heat is set free in the two phases of the reaction: first, in the condensation stage, by which the initial product is formed, and water is separated; second, in the final hardening when the product becomes infusible by polymerization, a considerable disengagement of heat takes place anew. If the substance be heated in thin layers, this self-heating may be unobservable on account of the heat losses, under such conditions; if, however, the mass is thicker or bulkier, and more especially if it be contained in a mold, this self-heating becomes very disturbing, and liberates gaseous or volatile products which cannot escape before the mass sets to infusibility; this causes the mass to swell and raise and to become porous, and makes it practically worthless for almost all technical purposes. This was the stumbling block which former investigators tried to avoid by conducting the hardening at very low temperatures or by the use of suitable solvents which tend to moderate the reaction. This tendency towards foaming exists also if acid-condensing agents are used, or even if no catalytic agents are added at all. The liberated gaseous products may vary according to conditions; in some cases they may consist largely of formaldehyde gas, which tends to escape before the reaction is accomplished; if ammonia be used, and more so if hexamethylentetramin be employed, varying amounts of ammonia gas will be set free.

Specially when hexamethylentetramin is used, the evolution

[24] Lebach, Zeitschrift angewandte chemie., 1909, p. 1600.

[25] The addition product of phenol and hexamethylentetramin. See Beilstein, Handbuch der Organischen Chemie, Third Edition, Vol. II, p. 651.

[26] Knoll Belgian patent, loc. cit., and Wetter (Knoll), British patent, loc. cit.

of ammonia is very abundant, and this naturally increases the tendency to foam and to give a porous final product. This tendency to foam becomes pronounced only at temperatures above 100° C., because at these increasing temperatures the exothermic reaction sets in. It should be noted that temperatures considerably higher than 100° C. are those which are employed in almost all commercial applications of these products, because they allow quick hardening and quick molding. At such high temperatures, polymerization proceeds very rapidly, but the exothermic reaction superinduces a further spontaneous increase of the temperature of the mass, and in this way the defect of foaming is considerably more pronounced.

This tendency to foam makes it of the utmost technical importance, whenever high temperatures are employed, for quick commercial work, that the liberation of gaseous or volatile products during the polymerization or hardening process should be opposed by a suitable counter-pressure. The latter may be applied in various ways; for instance, by heating in closed molds, or in closed vessels, so that the imprisoned gases develop a suitable counter-pressure; or by heating in a chamber in which air or other gases have first been pumped to a suitable pressure; or by heating in a hydraulic press. In the latter case, the first function of the pressure is to counteract the development of gaseous products, while at the same time, the mass is given the desired shape in the mold. Baekeland, U. S. Patent, No. 942,699.

For other applications, like varnishes or lacquer, where the material is applied in thin layers, the use of counter-pressure is not indispensable.

As stated above, the use of ammonia or hexamethylentetramin increases the tendency to foam. On the other hand, small amounts of fixed alkalies, like caustic soda, act as more powerful accelators than ammonia or hexamethylentetramin, without causing the evolution of disturbing ammonia gas or other gases. In this, and other respects, the fixed alkalies have decided advantages over ammonia or hexamethylentetramin, as well as over acid-condensing agents. For instance, they permit rapid hardening at the relatively low temperatures of 70° to 95° C.; furthermore, as soon as the initial solidification has set in, the temperature can be raised quickly to 110°, 120°, 160° C. At these higher temperatures, the hardening proceeds with great intensity and without fear that the gas bubbles should cause

porosity. If the heating be carried to the hardening temperature, before all the water has been first expelled, then the only necessary precaution will be to keep the temperature sufficiently below 100° C., so that no steam should be evolved, which might cause blisters; but as soon as the mass has been heated long enough at these lower temperatures, so that it has solidified sufficiently, the temperature can be raised with impunity above the boiling point of water. As soon as these higher temperatures become available, the polymerization to final hardening advances very rapidly.

For many purposes, it is simpler to drive off the water at temperatures below the polymerization temperature, either by drying in vacuo, or by drying in a stove at ordinary pressure at moderate temperatures, for instance, 50° C. or below. Such dried material can now be submitted directly to relatively high temperatures without risk of blistering or foaming. This gives us the very best means for rapid hardening, as required by commercial processes. The use of these fixed alkalies has enabled us to carry on hardening and molding at a faster rate than is possible with ammonia or hexamethylentetramin, or other means, and at the same time to produce molded articles of better heat-resisting qualities, of highest resistivity to solvents, chemicals, and of excellent dielectric properties. For many electrical purposes, the fact that no free ammonia exists in the mass, is a further advantage; indeed, this free ammonia is slowly liberated by heat from molded articles and sometimes may play rather disturbing pranks. It has a tendency to corrode brass articles.

Second Method. Action of Formaldehyde or its Equivalents on Phenol Alcohols.

I described this process in 1908.[27]

It has been shown that the best results are obtained if the amount of formaldehyde is at least one-sixth of a molecule, as calculated to one molecule of phenol-alcohol. This same ratio holds good if substances equivalent to formaldehyde or to phenol-alcohols are used.

[27] The Synthesis, Constitution, and Uses of Bakelite, loc. cit. See also Baekeland, Belgian addition, patent No. 213,576; Baekeland, French addition, patent No. 11,628.

This method has enabled us to gain clearer insight in the relations of all infusible condensation products to the phenol-alcohols, and has furnished us the theoretical means for determining the optimum quantities of reacting materials in our technical methods of manufacturing.

However, this process is more of theoretical than of practical interest, in as far as the third method accomplishes substantially the same result by starting from the anhydrides of phenol-alcohols.

Third Method. Action of Formaldehyde or Its Equivalents (Paraform, Hexamethylentetramin, etc.) on Saliretin-resins.

The method is another indirect method and consists in first preparing a saliretin-resin of the fusible soluble type, then reacting thereupon with formaldehyde or an equivalent of formaldehyde.[28] This method was first published by Lebach at the end of 1907.[29]

In these patents, it is clearly mentioned that paraform and hexamethylentetramin are equivalent to formaldehyde in the preparation of condensation products. Furthermore, it is described how the condensation products may be prepared in two successive steps by adding the formaldehyde or hexamethlentetramin, or other equivalents, in two successive quantities. Briefly stated, the process consists in first preparing a fusible saliretin-resin, then to this resin is mixed a second quantity of formaldehyde, paraform, or hexamethylentetramin; this mixture submitted to heat produces the infusible product "C." In reality, we prepare here, in two steps, a product which is practically similar to the solid initial condensation product described in the first or direct method. In that method, the initial condensation product is obtained more directly by the addition of a sufficient amount of formaldehyde to phenol, in presence of ammonia, or other bases, or by the equivalent use of hexamethylentetramin or paraform. In the first or direct method, the reaction between the phenol and the formaldehyde ensues under elimination of water due to so-called chemical condensation. In the present

[28] Baekeland, U. S. Patent No. 1,038,475, granted after interference with Aylsworth.

[29] Knoll, Belgian Patent No. 204,811, Dec. 31, 1907, and Wetter (Knoll), British patent 28,009, 1907, all owned by the Bakelite Gesellschaft, of Berlin.

case, however, a portion of the formaldehyde is first made to react on an excess of phenol, bringing about a corresponding elimination of water by chemical condensation; but in as far as the amount of formaldehyde is insufficient, fusible saliretin-resin is formed. In order to transform the latter into the product "C," it is necessary to supply an additional amount of formaldehyde, or some paraform, or hexamethylentetramin, etc; hence the necessity of adding a certain amount of those methylen compounds to the fusible saliretin-resin before the mass is submitted to hardening or polymerization by heat. The chemical reaction of the methylen compound on the fusible saliretin-resin is accompanied by the further elimination of water, which can easily be demonstrated by direct experiments.

If hexamethylentetramin is used, an abundant liberation of ammonia takes place at the same time; but even with the use of the latter, a certain amount of water is liberated by the action of the hexamethylentetramin on the free phenol contained in the fusible soluble saliretin-resin.

Barring those minor differences in preparation and proportions, the final product "C" is practically the same as what is obtained by the first or direct method as described above.

If hexamethylentetramin be added to the fusible resin, and heat be applied, the violent exothermic reaction which ensues causes an abundant liberation of ammonia gas. The mass raises like bread, and a hard spongy product is the result. Aylsworth[30] utilizes this foaming to prepare this substance in powder form by first producing porous masses of the final condensation product, which can be crushed more easily to a fine powder than if solid lumps of this refractory material have to be pulverized.

In molding processes where high temperatures are needed, so as to insure quick hardening, this violent liberation of ammonia gas can easily be counteracted by suitable counter-pressure. In this case, the pressure is not only required for shaping the article, but first and foremost, for avoiding porosity. This can easily be demonstrated by heating the mass in an open mold, at the same high temperature as is used in the press; under these conditions, direct application of these high temperatures causes foaming and porosity, unless suitable counter-pressure be applied.

It has been claimed that by the use of hexamethylentetramin in

[30] Aylsworth, Belgian Patent No. 240,116.

connection with perfectly dry fusible soluble phenol-resin, no water is liberated, which is supposed to be an advantage for certain applications where high dielectric properties are required. However, it should be noted that such fusible soluble phenol-resins all contain considerable amounts of free phenol and by the action of hexmethylentetramin on this phenol, water is formed besides the ammonia that is set free.

Moreover, the presence of large quantities of free ammonia is, if anything, more objectionable for certain purposes than the possible presence of small amounts of water.

It is true that free ammonia can be expelled by sufficiently long after-drying of the molded articles, but by the same means it is just as easy to expel the last traces of water. The most striking fact is that there is no serious difficulty in obtaining articles of extremely high dielectric properties, even when starting from raw materials containing considerable amounts of water, provided the manufactured articles be submitted afterwards to a drying treatment, which can be performed in any suitable drying stove.

It should be noted that almost all molded commercial articles made of phenol-formaldehyde condensation products contain various amounts of fibrous materials, preferably wood-pulp or finely divided sawdust. These organic fibrous materials, at the high temperature at which the molding in the hydraulic press takes place (140° to 160° C. or over), begin to liberate variable amounts of water and other products of decomposition, which depress the dielectric properties. This is another reason why all molded articles intended for purposes where high dielectric properties are essential, should be submitted to oven-drying after they are molded. Those who are unfamiliar with the technical side of the subject may ask why it would not be simpler to omit altogether the use of fibrous organic materials. They might suggest the use of asbestos. But asbestos has other drawbacks, which limit its use. For instance, asbestos is a relatively poor insulator, and the strength imparted by its fibres is not so great as that imparted by vegetable fibre; furthermore, its specific gravity makes the articles compounded therewith very heavy; moreover, any asbestos compositions which have to be machined or milled are very severe on the tools. Another objection is that asbestos compositions do not take the excellent and easy polish which can so easily be developed on articles made with

wood-fibre compositions, nor do they possess the elasticity and strength of the latter.

It might also be suggested to drop entirely the use of any fibrous material, and to use amorphous or pulverulent fillers. But the technical requirements forbid this. Indeed, the main characteristic of the final phenol-formaldehyde condensation products is that although they are exceedingly hard and resistant, and have a remarkably high crushing strength, their flexibility and elasticity are very limited. In regard to these latter qualities, they occupy a position between hard rubber and glass. A sudden shock or limited bending shatters them, and this would restrict enormously their technical applications. I found that the shattering wave induced by impact could be considerably modified by the suitable introduction of fibrous or cellular materials, like wood-fibre. This behavior is quite different from that of other plastics like celluloid or rubber, which lose their best and characteristic qualities by the incorporation of filling materials. The phenol-condensation products, on the contrary, are enormously improved for commercial use, if compounded with filling materials, provided the latter be of a fibrous nature; in the latter case, they lose their natural brittleness; can stand shock and impact, without shattering; they can be rendered flexible and yet maintain all their other excellent properties of high resistivity to physical and chemical agents.

Hence, some of the most important industrial applications of these condensation products are precisely those where they are used in conjunction with fibrous bodies; for instance, they serve to impregnate fibrous materials like wood, pulp-board, and to indurate the latter, or to agglutinate firmly loose fibrous substances, like wood-fibre, or fine sawdust, which then act as a structural skeleton distributed throughout the indurated mass.

This important technical result is easily demonstrated by comparing the enormous strength and resistance to impact or shock of molding compositions containing wood-fibre with others containing the same amount of structureless filling materials, for example, powdered materials. Compositions made with the latter will be found incomparably more brittle and very much less appropriate, if not entirely unsuitable for most industrial purposes, and more especially for molded articles where great strength is required.

Other important technical results are accomplished with the use

of filling materials; for instance, the highest dielectric properties have been rendered possible by the joint favorable action of fibrous material and heat and pressure.

It might be cited here that paper impregnated with these condensation products, and submitted to hardening under heat and pressure, has made it possible to manufacture sheets which show an astonishingly high disruptive test (puncture test), averaging 77,000 volts a. c. on sheets on one-sixteenth of an inch thick, corresponding to 1230 volts per mil or about 48,500 volts per millimeter.

Under these favorable conditions, the vegetable fibre of the paper is thoroughly impregnated with the condensation product, and the high pressure has excluded the possibility of porosity induced by foaming.

PROTECTION OF INTELLECTUAL PROPERTY IN RELATION TO CHEMICAL INDUSTRY

By President L. H. BAEKELAND

Read at the Detroit Meeting, December 4, 1912.

The mass of unthinking people, as well as those whose views are predominantly guided by precedent, have little or no conception of the natural rights of intellectual property. It is difficult to teach such people that adequate protection of intellectual property is abundantly more beneficial to the community at large than to the temporary individual possessors of these rights.

Yet these same people consider as sacred and inviolable any other property rights as soon as the latter relate to chattels or real estate, whether such rights were obtained by purchase, by inheritance, by gift, by privilege, by labor, or in any other way.

Furthermore, the laws of all nations are very strict in protecting such property rights, but do not concern themselves beyond certain limits, whether the possessor of the property is morally entitled to it or not. Neither do our laws concern themselves whether the owner uses his property for good or for wrong, for the benefit of the community at large, or for the gratification of his own selfish purposes. From the standpoint of the law (with very few exceptions, such as, for instance, Board of Health or police ordinances, or cases of so-called eminent domain), it matters little whether the private ownership of some property is a burden to the community or whether it is an impediment to the happiness or the free development of its citizens.

Neither is there any dispute as to the time the ownership of such property should last. Except for restrictions put on ownership by taxes, property rights are practically perpetual, and can only be transferred by accepted methods, as, for instance, sale, barter, inheritance or donation.

In some rare instances, there may be expropriation for public

purposes (or eminent domain), but even then, some suitable compensation is usually made.

All this is readily accepted as an axiom, as an underlying article of faith by all laws relating to property. Only the socialist dares dispute these rights, while even the single-taxer admits them to such a decided extent that he desires to abolish taxes on all property created by labor or enterprise, so as to shift the burden of all taxation on unearned land values.

When, however, it comes to recognize the claims of ownership to intellectual property, the result of the truly creative effort of the citizen, we butt right away against some stubborn conceptions, which have petrified into the code of our long-established laws.

If Tom steals Dick's two-dollar scarf-pin, Dick will have little trouble in putting Tom in jail, even if Dick himself has obtained his pin by questionable methods. But when it comes to protecting even for the short period of seventeen years, the most logical, the most legitimate personal property, intellectual property as embodied in patent rights, with all that it involves, with enterprises depending thereon, based often on the work of a lifetime, then our law courts are woefully deficient, on account of the uncertainties, delays and enormous expenses connected with the adjudication of patent rights. All this works overwhelmingly in favor of the litigant with the well-filled purse, the large corporation.

Yet no country in the world has expressed in a fairer and broader spirit the rights of intellectual property than the United States, in Article I, Section 8, of the Constitution: "Congress shall have power to promote the progress of science and the useful arts by securing for limited times to authors and inventors the *exclusive* right to their respective writings and discoveries."

This proclamation lifted the right of a patentee at once far beyond the mere privilege conferred by most other countries, which grant patents not only to the real inventors or originators, but also to those who are first to introduce unpublished inventions into their respective countries. With some legitimate pride, we can say that in this respect at least, American patent law stands head and shoulders above the laws of Germany, France and England.

The principles of the right of intellectual property so clearly defined in our Constitution, were repeated in the preamble of the French Law of January 8, 1791, which declares:

"The National Assembly, considering that every new idea, whose manifestation or development may become useful to society, belongs to him who conceived it and that not to regard an industrial invention as the property of its author would be to attack the essential rights of man; considering at the same time how much the lack of a positive and authentic declaration of this truth may have contributed till now to discourage French industry by occasioning the emigration of numerous distinguished artists and by causing to pass out of the country a great number of new inventions from which the Empire ought to have drawn the first advantages; considering finally that all the principles of justice, of public order, and of national interest imperatively command that it determine for the future the opinion of French citizens with regard to this class of property by a law which consecrates and protects it, . . . etc."

The wisdom of these provisions has been abundantly proved by subsequent events. Only a man stubbornly blind to evident facts will deny that just those countries which have the most liberal laws for patent protection, are also those which have taken the lead in the industrial and scientific development of the world. No man was more imbued of the benefits of the patent system than Abraham Lincoln, when in 1860, in his speech at Springfield, Illinois, he said:

"In the world's history, certain inventions and discoveries occurred of peculiar value, on account of their great efficiency in facilitating all other inventions and discoveries. Of these were the art of writing and of printing, the discovery of America, and the introduction of patent laws. . . . The patent system . . . added the fuel of interest to the fire of genius, in the discovery and production of new and useful things."

Up to about thirty years ago, our patent system covered tolerably well the purpose for which it was intended. It stimulated individual inventions and promoted numerous private enterprises. Since then, with the extraordinary growth of our nation, with the tremendous increase of agglomerations of capital for industrial enterprises, and more specially with the astonishing increase in the ramifications of applied science, our patent system has become totally inadequate to the needs of the country; it suits our new conditions in about the same way as baby clothes fit an overgrown boy.

Our patent system, although based on an excellent fundamental law, has now degenerated into a set of exceedingly complicated

technicalities of law practice, a system of legal acrobatics, whereby any contestation before the courts can be turned into "perpetual motion" to the advantage of wealthy litigants, and whereby the individual patentees of slender means and the small industrial concerns, find themselves under smothering disadvantages when opposing rich antagonists. In this way our patent system, instead of accomplishing its intended purposes of stimulating individuality, simply reinforces the rich and big industrial enterprises, and discourages the individual inventor unprovided with a liberal bank account.

I shall not take up your time by repeating all that has lately been published on the subject, but refer you to the available printed publications: Abuses of our Patent System, L. H. Baekeland, Journal of Industrial and Engineering Chemistry, Vol. 4, p. 333, 1912; The Incongruities of Patent Litigation, ditto, Vol. 4, No. 11, November, 1912. The United States Patent System, Robert N. Kenyon, Transactions of the American Institute of Chemical Engineers, Vol. IV, 1911. The Gist of the Supreme Court Decision in the Dick Patent Case, and of the Proposed Law Amendments, Gilbert H. Montague, The Engineering Magazine, May-June, 1912. The Sherman Anti-Trust Act and the Patent Law. The Supreme Court on Patents (the Dick Patent Case), Gilbert H. Montague, Yale Law Journal, April-May, 1912. Report No. 1161, to accompany H. R. 23,417, August 8, 1912, Hon. W. A. Oldfield, Chairman of the Committee on Patents, Washington, D. C.

It is true that on November 4, 1912, the Supreme Court of the United States promulgated revised Rules of Practice for the Courts of Equity, which intend to simplify our methods of litigation. Unfortunately this is only a half-way measure, leaving still abundant opportunity for the tactics of delay, chicane, and expense which have too much disgraced American patent litigation.

These new rules might gain in efficiency, if they were supplemented by the creation of a final court of patent appeals. They might be made incomparably more efficient if they could be strengthened by a system whereby the adjudication of the validity of patents does no longer devolve upon judges who do not possess the technical or scientific preparation required nowadays for discerning the merits of complicated patent questions. Some of the far-reaching details of scientific technology absolutely baffle the

comprehension of those who have no preliminary technical or scientific training. Certain problems of chemistry and physics involved in many patent suits can no longer be understood by an intelligent judge, if he has not had long and systematic preliminary training in that branch of knowledge. I do not deny that an intelligent judge can be coached and instructed by long, tedious, time-robbing methods, even in intricate scientific problems; but his education has to be made over again for each special case. After you have made a chemist of him for one case, the next adjudication will require the knowledge of a physicist, an electrician, an engineer, and so forth.

What would any judge say of a chemist or a mathematician, or an engineer, totally ignorant of the practice of law, who tried to conduct a law case in court? Such an amateur lawyer might succeed in doing so, but to what hopeless loss of time, misunderstandings and confusions would this lead before the subject had been mastered to some extent? Yet this is exactly what happens with a judge to whom we entrust to decide on the validity of a patent involving highly intricate scientific or technical subjects.

Judge Hand expressed himself very eloquently on this subject:

"I cannot stop without calling attention to the extraordinary condition of the law which makes it possible for a man without any knowledge of even the rudiments of chemistry to pass upon such questions as these. The inordinate expense of time is the least of the resulting evils, for only a trained chemist is really capable of passing upon such facts, e.g., in this case the chemical character of Von Furth's so-called 'zinc compound,' or the presence of inactive organic substances. In Germany, where the national spirit eagerly seeks for all the assistance it can get from the whole range of human knowledge, they do quite differently. The court summons technical judges to whom technical questions are submitted and who can intelligently pass upon the issues without blindly groping among testimony upon matters wholly out of their ken. How long we shall continue to blunder along without the aid of unpartisan and authoritative scientific assistance in the administration of justice, no one knows; but all fair persons not conventionalized by provincial legal habits of mind ought, I should think, unite to effect some such advance." (See Parke-Davis & Co. *vs.* M. K. Mulford Co.,

Circuit Court, Southern District of New York, April 28, 1911, 189 Federal Reporter, 95.)

Even under the new rules it will not be difficult to drag on a case by presenting an unrestricted amount of testimony taken before an incompetent examiner and by calculating every step so as to tire out your opponent, and so as to lead the judge into doubt and error, by swamping him with endless contradictory expert testimony calculated to befog the issue instead of making it clear. Such tactics are relatively easy for the litigant who, for that purpose, can afford to pay accommodating experts and skillful lawyers. Even if at the end the judge, after laborious and conscientious efforts, masters the technicalities of the case and reaches a good decision, much needless time has been wasted. All this might easily be avoided, and judges might be saved the trouble and responsibility of going in every single case through a different scientific or technical training, if their intervention could be limited to what they are competent for, namely, to determine what claims have been infringed and in how far this infringement entitles the patentee to damages.

That such a method of settling patent suits is quite practical, is shown by the example of Germany. In that country patents are allowed after preliminary examination, just like here; but, after the patent is granted, it can be attacked for annulment or revocation before a competent court in the Patent Office. So that any party who is sued for infringement of a patent which he thinks is invalid, can avoid temporarily the adjudication of the infringement issue by starting an annulment or revocation suit. In the meantime, the courts in which infringement cases are examined have to take the patent as it stands, and it is only left to them to interpret the scope of the claims, and to what extent these claims have been infringed.

This relieves the equity court of all the complicated questions of validity or non-validity of a patent, and puts the whole matter in the hand of a properly constituted court of experts, who can handle this subject with incomparably less hesitation or delay. Besides this, the whole system of practice in the German Patent Office tends toward systematic elimination of invalid patents. After an examiner has decided upon preliminary allowance of a patent, the claims and specifications are open for public inspection, and for a

period of two months anybody whomsoever can file arguments against the final grant of the patent. In this way, the nation does not confer too lightly patent privileges and has, furthermore, the benefit of the free advice of any experts in the art, who may advance good reasons for non-allowance of the claims, of which the examiner was not aware when he rendered his first decision. These opposition proceedings give added thoroughness to the work of the examiners. They are relatively inexpensive and do not necessitate the intervention of law counsel. Sometimes they delay the issue of a patent, if there is any good reason for doing so. On the other hand, a patent that has successfully withstood vigorous opposition proceedings is very much strengthened thereby. This, in itself, is a very valuable compensation for any delays to which the patentee may have been subjected. In other words, by that system, a good patent becomes stronger, while a defective patent application is easily weeded out. A similar system of public opposition exists here in the United States in relation to the granting of trade-mark rights, and seems practical enough to be extended to our methods of allowing patents.

Such a sifting process, first by the examiner, then by opposition proceedings, sometimes by annulment or revocation proceedings, for wrongly issued patents, involves no serious difficulties nor great loss of time if carried out by courts of experts. Thanks to such a system, the work of a judge who acts on an infringement case, gains considerably in dignity and is, at the same time, enormously shortened and simplified. (See Wertheimer, The German Patent System, *Electrical World,* May 18, 1912.)

The German system throws the burden of technicalities and expert knowledge on the Patent Office, or the courts connected therewith. Nothing would be easier than to introduce a somewhat similar system in our country.

All officers of our patent office, high or low, should be made independent of any political favoritism; they should be better paid, with more opportunity for promotion, according to merit; their work should be made simpler by an improved office equipment and increased facilities for a thorough search; furthermore, our unnecessarily complicated and expensve methods of interference proceedings should be simplified.

With these reforms, there is no doubt that we can organize right

in the Patent Office, a competent court, supplemented by the court of appeals of the District of Columbia, for deciding in a very expedient way all questions of validity of patents.

This court of appeals, because it is situated right in Washington, would have easy and immediate access to all the records of the Patent Office; by this fact alone, it would have superior opportunities for prompt and efficient work.

During recent years, Germany has been trying to broaden its patent laws more and more towards the principles set forth in the American Constitution. For instance, it has practically eliminated the system of compulsory licenses except in some rare instances where public welfare is involved. If only we could borrow some of the more efficient methods with which the German patent law is administered, and enforced, we might succeed in making an American patent *real* property for poor and rich alike, instead of a pretext for expensive and endless litigation, with all the advantages it gives to the richer litigant, to the detriment of the consumer, who in the end pays the bill.

At least some of these facts seem to have been very well recognized in the masterly report of Hon. William A. Oldfield, chairman of the House Committee on Patents. (See report No. 1161, on H. R. 23,417, August 8, 1912.)

Unfortunately, his proposed Oldfield Bill (H. R. No. 23,417), with a regrettable lack of consistency, neglects utterly the paramount issues, and busies itself with secondary regulations, which, if carried out, will practically put a penalty on patented articles.

The new provisions of the Oldfied Bill aim at curtailing the power of patents in the hands of trusts or large corporations; but, in doing so, new provisions are introduced which will create endless new opportunities for protracted litigation.

The Oldfield Bill overlooks the axiom that whatever increases the expense or delays of litigation is a very potent weapon in the hands of large corporations, which they can hurl against the poor litigant who stands in their way.

The saddest thing of all is that the new Oldfield Bill tries to abrogate the hitherto accepted principle established by our Constitution, that the patentee has the right to license or sell his patent on whatever terms he pleases. It has been feared that this principle, if carried too far, might become a dodge for avoiding anti-

trust laws. Since the decision of the famous, but harmless, Dick case, the most hysterical exaggerations have been published on this subject. Fortunately, since then, the recent and unanimous decision of the United States Supreme Court in the "bath tub trust" case, November 18, 1912, does away with all these redundant arguments and settles, beyond doubt, the principle that, patent or no patent, unlawful combinations in restraint of trade can be stopped by the Sherman Law.

The Oldfield Bill, in its eagerness to avoid any hesitation on this subject, goes one step further, and unfortunately, one step too far. It puts so many restrictions on the sale of a patented article, or on a patent license, that it may become a positive disadvantage to transact business by means of patents.

Examined in its last analysis, it threatens a business based on patented processes or patented articles, with penalties which unpatented articles are thus far not subjected to. It takes the proposed patent law as a pretext for saddling a patented article with restrictions which have not heretofore been formulated for non-patented goods.

This unexpected paradox, promoted by the Oldfield Bill, is distinctly in opposition to the rights of intellectual property conveyed by the words and the spirit of the Constitution, and if the Oldfield Bill becomes an effective law, it will be the saddest blow ever given to our patent system. It will do comparatively little harm to large business interests, because for them, there are many ways of circumventing its provisions; on the other hand, it will cause great discouragement to smaller enterprises which until now have held the hope of matching inventive genius and initiative against the money power of big organizations. Make a large corporation respect the patents of a small concern, or of an individual, and you reduce at once any advantage of size or money power, and at the same time, you encourage the most beneficial form of competition, competition based on improvements. But to introduce curtailing restrictions for the licensing or selling of patented articles or patented processes to which non-patented articles are not subjected, means simply obliterating the value of patents while needlessly increasing still further the opportunities of endless and ruinous litigation and chicanery.

Another unfortunate miscarriage of purpose in the Oldfield Bill

is its provision against so-called wilful "suppression" or "non use" of patents. It does not take into consideration that in numerous instances, a patentee or an assignee possesses a series of so-called alternative patents, which can be used to bring about identical or similar technical results by modified means. Among such alternate patents, the best or the most suitable is used, absolutely irrespective of any other reason or intention to suppress their use. Yet without the exclusive possession of every one of these patents, the invention would not sufficiently protect against competitors, and the field would be so much reduced as not to make it worth while to put one's best energies to the development of the invention. In most cases, it would become a material impossibility for a small concern to maintain the exclusive ownership of its patents, if it had to go to the enormous expense of working simultaneously all its "alternate" patents; by omitting this expensive technicality, it would be exposed to the risk of being compelled by its competitors to grant a compulsory license; this would practically annihilate the advantage of exclusive ownership as expressed by the Constitution. Here again large concerns would be at an overwhelming advantage because they can at an expense relatively small for them, equip the necessary appliances for remaining within the technical provisions of the law. In the meantime, they could easily harass their financially weaker competitors by exacting from them compulsory licenses which would break up the only prospects of successful competition which the smaller concern might have possessed, until then, in its patents.

In other words, the Oldfield Bill is aiming at the petty side of the situation, and in doing so, has unwittingly picked out a vital spot of our patent system. It reminds one of the man who set his barn on fire in order to drive out a hornet's nest.

I have no doubt that this bill has been framed with the best intentions for the interests of the country. Unfortunately, the framers of this bill do not foresee the far-reaching and dangerous effects of its provisions.

The average man, even the average legislator, has a rather one-sided conception of patents or inventions. Most people's idea of a patent does not go far beyond some simple mechanical device, like a patented mole-trap, a safety razor, an alarm clock, or other similar invention, more or less easy to understand after the apparently

simple mechanical principles have once been explained. Then everything seems so simple and easy to them, that their limited imagination cannot conceive how even these apparently simple devices have frequently cost incredible efforts and immense amounts of money before their advantages become available to the public.

This attitude of mind develops, naturally, the belief that a patentee has a "soft snap," the result of a lucky idea, in about the same way as a lucky prospector strikes a rich gold mine, or a lucky ticket draws the grand prize in a lottery.

Precisely on this account, it becomes difficult to explain to such people the rights and purposes of intellectual property; it is still more difficult to convince them that the nation is greatly benefited by liberal patent laws.

When it comes to chemical patents, the ignorance of the average public is amusing if not pathetic. Since we have heard a New York alderman in an official address of welcome to the members of the International Congress of Chemistry speak as if they were druggists or pharmacists, we must no longer be astonished if the average Congressman or Senator refers to a chemical patent as a synonym of "patent medicine."

But it is even difficult for the better prepared legislators, to understand how some chemical inventions have brought about the most far-reaching developments, not only in other industries and arts, but in civilization itself.

For instance, it is not so obvious to them how processes for fixing the nitrogen of the air, or extracting soluble potassium salts from rocks, enable us to make food supplies independent from the restricted potash mines in Germany or the nitrate deposits in Chili. Such inventions are no more nor less than a means for preventing possible starvation of our race. Do they realize that the development of the automobile, with all that it directly and indirectly implies, was entirely dependent on Goodyear's vulcanizing process of rubber? Shall we remind them of the fact that without the invention of explosives, like dynamite, gigantic engineering enterprises, the Panama Canal, blasting of rocks for the excavation of our cities, mining for ores, tunneling and grading of railroads, would be impossible? How could we expect even the most perfected modern printing presses to distribute to every citizen, rich

or poor, young or old, that knowledge and culture, which means better citizenship, better opportunities for happiness and development of our race, if it were not for the inexpensive and abundant supply of paper furnished by the cellulose processes. The Greeks, the Romans, and even the Middle Ages, had their sages, their poets; yet those were the times of slavery and oppression, because knowledge was only in the reach of such a limited number that it was possible for tyrants to throttle its diffusion by sending the few advanced thinkers to the gallows or burning them alive. For the same reason, scarcity of books, the destruction of the library of Alexandria, was a calamity for the intellectual development of mankind. Our abundant supply of cellulose makes a repetition of such conditions an utter impossibility.

Then again, where would we find our supplies of steel, the main raw material for modern engineering, if the Bessemers, the Thomas-Gilchrists and others had not invented their processes? How about the marvelous synthesis of products derived from coal tar, which have literally created the most astounding series of new substances which have revolutionized therapeutics, surgery, hygiene, and are finding daily new applications in the most varied arts in general technology?

At a time when all countries are confronted with that critical question of the increased cost of living, it may be interesting to point out that just those industries where invention and patents have played the smallest rôle, are also those where the increase of price is most burdensome, while those commodities where patented inventions have had the fullest influence, have, on the contrary, decreased in price, and in some instances, to an astonishing degree.

For instance, the price of sulphuric acid is about fifteen times less than it was in 1807, and about one-half of that of 1870. The price of soda ash is about one-sixth of what it was in 1823, and about one-half of the price in 1860. Nitric acid sells for less than one-half the price of 1861. Glycerine sells for about one-eighth of the price of 1855. Chloride of lime in 1800 sold for 30 cents a pound, in 1870 for about 2 cents per pound, to-day for about 1 cent a pound. Any chemist knows that every one of these products is used directly and indirectly in the most ramified channels of our arts and industries, but the layman does not know that cheap soda means cheap soap, cheap paper, cheap glass, etc., that cheap sul-

phuric acid means cheap fertilizers, better crops, cheaper corn, cheaper wheat, and so forth.

Let me point out that the decrease in price of these materials is even considerably greater than the bare comparison of figures indicates, if we take into consideration that the purchasing value of money has considerably decreased, while the cost of labor has enormously increased.

Nor are these examples merely confined to chemical products. The reduction in price for articles where patents have played an important rôle is just as evident in steel products, tools, machinery, etc.

Compare these lower prices with the vastly increased cost of rents, clothing, foodstuffs and many agricultural products, where patents have played a less preponderant rôle. If you will carry your analysis still further, you will find that in such branches of trade where patented inventions have had little or no importance, for instance, cattle raising, prices have soared highest. On the other hand, for such agricultural products where patented machinery could be used to best advantage, like wheat and corn, the increase of price has been relatively small. Then again, garden vegetables, potatoes, etc., where the use of patented agricultural machinery is less available, show an enormous increase in price.

You may object that the price of shoes has gone up, but here again, the increase is entirely due to the greatly advanced price of hides, and were it not for the perfected shoemaking machinery, and for the better and cheaper chemical tanning methods, all due to patents, the cost of our shoes would be so high that they might again become an article of luxury, available only for the well-to-do.

The present price of clothing is high enough as it is; nevertheless, it would still be much higher but for the patented machinery for spinning and weaving, the patented chemical processes of bleaching, dyeing, mercerizing, etc.

I should not omit to mention our vastly improved and cheaper methods of transportation, of production of power and light, all developed and perfected on an interwoven system of patents. I could explain the far-reaching influence thereof on civilization, culture, on the happiness and security of life of the individual citizen; but even then I might not convince the pessimist or the

scoffer, who only sees the hole in a doughnut and stubbornly persists in ignoring the doughnut itself.

The history of almost every invention which we are utilizing now, unconsciously, every day, is an epoch by itself, the details of which are only known by the few pioneers who gave the best they had to give, who helped with their brains, with their money, and talent of organization; some with their very lives.

The oft-repeated statement has been made: "An inventor cannot help inventing, whether you give him a reward or not." Then again, some others say: "Necessity is the mother of invention."

The most apparent fact is that the man who receives an ample income from his father, or some other privileged source, is less prompted to distinguish himself by arduous creative work on inventions than the poor but intelligent man who sees in invention a means of making himself financially free and independent, as well as giving an outlet to his inventive abilities.

Whoever has followed intimately the development of some chemical processes knows very well that whether "the inventor cannot help inventing," or whatever may be the incentive to invention, most of these important inventions could never have been carried out, or could never have been brought to the point where they became of public benefit, but for the intelligent use of vast sums of money. Too few people have a conception of the immense sacrifices, of the serious money risks, involved in the development of some patents. Many chemical inventions used now currently and open to the public at large, have cost millions before they were brought into practical shape, or before the public was educated to their advantages. Can any one expect that such expenses, such efforts, such risks, would be undertaken, unless there was the possibility of at least some chance of recouping by a temporary patent protection?

Let us take, for instance, those large German chemical companies, which employ hundreds of chemists and engineers, engaged exclusively in research work; to them we owe the development of many processes which have had an untold beneficial influence in many directions on the economics of our daily life, even on civilization itself. They employ large aggregations of capital, reaching into many millions. The dividends of some of these companies may appear large to the superficial observer. Yet if you look more

closely into it, you will find that these very companies were founded long ago, some of them over half a century or more, that the large capital which they employ has never been "watered," that although they have had the benefit of the devoted cooperation of an endless number of distinguished men, stars of first magnitude in their profession, the net returns on their invested capital, at the end of half a century of brilliant intellectual pioneer work, is relatively small, even if the dividends seem large. In fact, the net returns are decidedly lower than that of many American enterprises not over fifteen years old, and where progressive technical leadership was entirely lacking, but where tariff privileges and agglomeration of competing concerns into a trust insured a splendid paying monopoly, notwithstanding the reckless financiering of their promoters.

If you will further investigate the history of those German chemical concerns which have become leaders of the industrial world by nothing but their intellectual pioneership, you will find that, notwithstanding all the patents on which they have to rely, the expenses involved in research work and pioneership, swallow up, to a large extent, the profits realized in some of the established branches. But with true scientific spirit, their far-sighted directors were willing to sacrifice a very considerable part of their earnings, in their search for improvements and development of new ideas; they have set a magnificent example in the only competition beneficial to the public, competition by improvement.

One of our wealthiest retired multi-millionaire manufacturers, not so long ago, speaking about his money successes, gave the following advice: "Never be a pioneer; it does not pay. Let the other man do the pioneering, and then after he has shown what can be done, do it bigger and more quickly; but let the other man take the time and the risk to show you how to do it." To anyone who advances the statement that an inventor "cannot help inventing," I desire to ask whether an inventor will do much inventing, if in order to carry on his research work, or to develop his invention, he has to spend hundreds of thousands, nay sometimes millions of dollars, but does not possess them, and nobody is willing to take the risk to furnish the money unless there is a fair chance for his backers of obtaining some compensation by a temporary patent protection? Those who know the large sums of money which have been swallowed up by the research and development work con-

nected with the artificial production of nitrates; with the Solvay soda process; the development of the steam turbine; electric light, electric traction, and numerous other inventions of far-reaching magnitude, will know what I mean.

Just on this account, it is highly unreasonable of the Oldfield Bill to try to make a distinction between the inventor in whose name the patent is drawn, and the party who runs the risks in enabling the inventor to make the invention available to the public. Any such legislation simply tends to discourage those who, at considerable risk, furnish the capital and the talent to develop an invention into a commercial possibility, and who thereby bring it into real public service.

Now and then, I have perceived that some of my fellow chemists, who, although highly trained, have never created anything of technical value, and whose experience with matters of practical life frequently does not extend beyond the confines of their lectureroom or their laboratory, do not seem to grasp fully the immense distance that lies between the initial conception of an invention, or its study in the laboratory, and the overwhelming amount of careful work and money risks connected with its development on a commercial scale, until it has safely reached the point where the public can avail itself of the invention.

I wish to cite, for instance, the famous Solvay process, which gives us cheap, excellent and abundant soda, an article of prominent importance in the wheels of our civilization. This process was known and described more than a dozen times, and had even been tried repeatedly at considerable loss, on a commercial scale, many years before Solvay tied his genius to this difficult problem and developed from an unreliable laboratory reaction a process of great industrial importance; then, with a staff of able collaborators, and the employment of large amounts of cash, he overcame, by and by, the technical drawbacks which had caused the failure of all of his predecessors.

Hundreds of similar examples could be cited. Whoever has been intimately acquainted with the commercial development of some of the most successful inventions, knows quite well the risks, dangers of failure, which have accompanied the herculean task of development and educational work. It is a well established fact that the great majority of new enterprises fail, that few succeed.

The educational effect due to the introduction of patented inventions is of immense benefit to the public, although this fact is not very apparent to most people. In many instances, the owner of a patent frequently has to go to extreme sacrifices before he succeeds in convincing the public of the merits of his invention; in fact, the public stubbornly refuses to benefit by an improvement to which it has not been fully educated.

The practical value of cash registers only became obvious after a most thorough and very expensive educational campaign.

The metric system is just as useful as the cash register; it was invented long ago and systematized in all its details during the first French republic. Nevertheless, to-day there are still two large commercial countries, the United States and England, which have not yet been educated to its merits; if the metric system had been patented, like the "cash register," somebody, during the seventeen years of the patent monopoly, would have undertaken the money risk and arduous task of thoroughly explaining the advantages of the metric system to our conservative citizens, and we would have ceased long ago to submit to the burden of waste of time and money caused by our antiquated, cumbersome system of weights and measures.

It has been stated, with much reason, that the best way to postpone the benefits of an invention is to allow public use of a patent, because then nobody takes the risk of starting an educational campaign or of developing the invention, which, after all, means pulling the chestnuts out of the fire for the benefit of others.

Entirely new industrial enterprises are not easily started on inventions which are not patented, unless some other method is available for insuring some kind of a monopoly; for instance, by maintaining secrecy or by acquiring special skill, or by controlling the raw material, or by tying the market, or in other instances where the initial outlay for a plant requires a capital so large as to exclude others.

Moreover, if you scrutinize those industries where secrecy of methods, instead of published patents, is the prevailing tendency, you will find that the secret-process-industries are precisely those which have least progress to record, and where high prices rule.

Whoever desires to get posted on the modern literature pertaining to any industrial chemical processes, will find that available

text-books are many years behind in information as far as novelty and accuracy are concerned; for this reason alone, it is absolutely indispensable to get acquainted with all recent patent literature.

Were it not for the compensation expected from patent rights, most of this information would be carefully kept secret, or if it were divulged at all, this would mostly occur by accident. Every newly published patent sets to work the thinking cells of numerous inventors, who are not slow to suggest further possible improvements. Every patent of some importance is rapidly followed by a succession of other patents conceived by other inventors, who were inventors, who were inspired by their predecessors, and so the work of progress goes on unceasingly and at a quickened pace.

In the age of the alchemists, there were no patents; inventions and discoveries were jealously guarded and buried with their originators, and the world and its inhabitants remained very much what they were, with most rights and comforts in the possession of those in power, and very little chance of improvement for the non-privileged classes.

The public should be educated in these truisms. Unfortunately, the education of the public has been directed in the opposite way since patent infringers have utilized the daily press after the late decision of the Supreme Court in the Dick case, to start a campaign for urging our well-meaning but ill-prepared legislators towards patent reform, which will give still broader scope to our modern buccaneers. This reminds me of the man who, after stealing a stranger's pocketbook, kept on shouting "stop thief," so as to distract the attention from himself.

Two ways are open for our legislators:

One way is to try "to hit the trusts" by mutilating the best there is in our patent system, which has been such a potent factor in the development of our country; to chill the best incentive for private enterprise; to stunt that kind of competition most beneficial to the public, competition by improvement, incomparably better in this respect for stimulating industry, science and progress, than protective tariff privileges which, in many instances, have worked in the opposite direction.

The other way is not to put dangerous restrictions on the patent rights defined by our Constitution. If there has been any fear that such patent rights might be abused for evading the provisions

of the anti-trust laws, these apprehensions have vanished by the clear unequivocal decision of the Supreme Court in the Bath Tub case.

But there is urgent need of reform in our patent system by simplifying procedure in the Patent Office as well as in the courts, by insuring better, quicker and less expensive means for adjudicating the title and validity of patents. Only such a reform will bring about that, big or small, poor or rich alike may be stimulated by the advantages of our patent system, instead of making a patent an expensive but powerful instrument, available only to the wealthy.

Whatever simplifies and lessens the cost of the administration of our otherwise excellent fundamental patent law, gives the enterprising man with small means a better chance of competition by inventive progress and merit against ponderous aggregations of capital. By such reform, which insures such healthy competition, the nation is sure to be benefited.

In all above considerations, my remarks were principally inspired from the standpoint of chemical patents, not alone because this very important class of patents is least understood by the average public and the legislator, but because chemical process patents are also those which are most difficult to protect from infringers.

Discussion.

V.-Pres. Whitaker: We have all heard the earnest and masterly address of our President. I think, as a rule, presidential addresses are not discussed, and usually for obvious reasons, but I think here is something in which we are all very much interested, and we should be very glad to have additional remarks and questions, if such occur to you in connection with this interesting subject.

Dr. Ittner: This patent question is a complicated one, and I confess that I do not feel competent to suggest the improvements that seem to be necessary—not nearly so well as Dr. Baekeland, because he is in a position to recognize things better than I am. I have seen the patent situation from perhaps a little different point of view from that which Dr. Baekeland has dwelt upon, although he has probably seen it from this point of view also.

I think that real inventors should certainly be protected in the rights to their discoveries, and that the right to intellectual

property should be respected. Now, I think that a person has a right to intellectual property, whether he makes that intellectual property public knowledge or whether he keeps it secret. There are some people who seem to think that whenever a thing is kept secret that the knowledge is piracy, but it is not always piracy, because the fact that it is pirated is evident in a product that is turned out, and sometimes in the product that is turned out there is no evidence of the process which is used, and for that reason there are some concerns who believe that they get better protection by keeping their processes secret, and I think that they have a perfect right to do so, and I think when they have property which they have developed themselves they have as much right to that property as those who make their property or their processes public knowledge. I think there should be some way of protecting those who have such processes, from being deprived of that right by those who come along later and possibly by delving into their secrets obtain those processes and even try to deprive them of their right to carry out the processes which they have discovered.

When a man discovers something new he does not know all the possibilities of it, of course. He may get some great principle which is new and very valuable and has great possibilities, but he does not know all the possibilities. He is the one who, in all probability, deserves the greatest credit which will come from that invention, and who should, I think, reap most of the benefits from it, but it is frequently the case, and it is my belief, that there are men who make a business of watching patents and studying the claims without being any great inventors themselves, or without having any great inventive genius, who study the claims of the patent and find defects or omissions. In fact, those omissions may be things which it is almost unnecessary to mention. Any man of intelligence would consider that they are too obvious to mention, and he does not mention them, but some man comes along and by mentioning these same things gets a patent on them. He seeks even to get a patent which would deprive a real inventor of carrying out his process, and sometimes he succeeds in doing it. I do not know how that can be righted. I believe that if a man has a process which is new, and if he can prove that he had that process and was carrying it out successfully before some one patented it, he should have the right to it.

President BAEKELAND: The point of view developed so well by Dr. Ittner is taken into consideration by the patent system of the United States. Dr. Ittner is perfectly correct when he contends that intellectual property should belong to the originator, whether the latter desires to patent it or to keep it secret. However, this country has devised the patent law as a way of making a "dicker" with the man who has a secret process. The nation says to him, "If you will divulge your secret we will give you a monopoly for seventeen years, but after that time, we confiscate your monopoly, and then your invention shall belong to the public." This sounds very well in theory, but in practice, the nation does not provide the protection which was promised to the inventor, and the practice of our patent system in the protection of patent rights is so difficult, and leaves so many loopholes to the infringer, that the patentee in return for the disclosure of his invention, practically gets a "gold brick" from the nation, under the shape of a patent certificate, which can only be enforced by wealthy people.

In theory, again, the American patent system provides for the case explained by Dr. Ittner, where a process is kept secret for some reason or another. For instance, it may happen that an inventor does not possess the money to take out a patent, or much less to defend his patent rights, if he had a patent; therefore, he may think it preferable to keep his process secret. By doing so, he may run the risk that his secret may be divulged. At any rate, he has a good chance that instead of seventeen years' protection, he may extend his monopoly for an indefinite time, not limited by law, and only limited by the care with which he guards his secret. Or again, a man may have invented a secret process, but may not think it worth while patenting, or he may think no patent could be obtained on it; or, what happens frequently, he may have tried to obtain a patent; and have encountered an examiner who thinks he knows everything and has decided that the subject matter is not patentable, and who on this account may have rejected his claims. In the meantime another inventor, who has employed a more convincing patent attorney, may have succeeded in obtaining a patent for practically the same subject by formulating his claims somewhat differently. In a case like this, the first inventor can, even after the patent of the second inventor is published, file a new application and obtain an interference. If he can prove beyond doubt that he is really the

original inventor, that he has pursued his work on this invention diligently and continuously, that he has not merely taken up the subject at some time, then dropped it, then taken it up again after the value thereof was demonstrated by the recent patentees; if, furthermore, he can prove that the subject matter has not been published for more than two years, either as a patent or in some other publication, and if the process has not been worked commercially for more than two years, he can still obtain a patent, which may in fact, be entirely similar in wording and in claims to the patent of the other patentee. Although the two patents may be coexisting, the only valid patent will be the one for which, in interference proceedings, priority of invention has been shown. All this sounds logical just as far as the first inventor is concerned. Unfortunately, these interference proceedings are frequently very expensive to both litigants, and may be complicated by appeals and reappeals and motions to dissolve, and the patentee with the slender purse is again at a tremendous disadvantage. In some cases, it is very difficult to furnish evidence, and in more than one instance, interference proceedings have been dragged out for many years and have cost the litigants more than hundreds of thousands of dollars before the patent was issued.

Other countries, for instance Germany, arrange this much simpler. The only date of priority is the date of filing the patent, as long as the invention is not known or published. This, of course, puts a premium on the man who files first his patent. After all, that is what the nation cares about: to have the benefit of an early disclosure of the invention.

Furthermore, our interference system has another serious drawback. For instance, you may have obtained a patent in good faith and feel entirely secure on account of it, and on the strength of your patent, you have started an enterprise in which you put all your own money, as well as the money of your friends. You go through all the worry and difficulties and the risks of pioneership, and finally, you succeed in convincing the world and the consuming public that your invention is really a good thing, and just at the moment when you are beginning to reap the reward of your enterprise, a man steps in with an interference, which has been kept smoldering in the Patent Office for several years. A new claimant arises, who jumps at your throat and says, "Your patent or your business life,"

and drags you in endless and expensive interference litigation, where the party provided with much cash and all that goes with it, is at an overwhelming advantage. If he succeeds in substantiating his claim to priority, your patent will simply become invalid. This gives opportunity for legally murdering a new enterprise. In fact, the principle of our interference system is such that practically anybody who starts any new industry, whether patented or not, and which involves any process which is not so hopelessly old that it is known all over the world, runs the risk that at any time somebody may jump at him with an injunction on the strength of some long-delayed patent application which has been "sleeping" in the Patent Office. A striking instance of this has been given by the famous Selden case, which involved the broad principles of automobile construction.

In Germany, such absurdities cannot occur. The spirit of the German patent law is very simple. It admits that the man who discloses his invention by taking out a patent confers a benefit upon the nation by becoming the teacher of the nation. Therefore, the nation is willing to grant him patent protection for a certain number of years. If, however, somebody has been carrying out the same process secretly before the patentee filed his patent, the latter can apply to the court, and if he can prove his case, the patentee may be compelled to grant a free license for the personal use of the other inventor, who has first exercised this process in secret in his own business. Indeed, in a case of the kind, the Germans reason as follows: If a man already knew the process and was utilizing it secretly in his business, but was not using it publicly before the patent was filed, he received no benefit by the publication of that patent. Therefore, he acquired the right of utilizing the process, undisturbedly, at least for his own purposes in his own factory. By keeping his process secret and by not filing a patent, he forfeited, however, any claims to national protection under the shape of exclusive patent rights.

Our American patent system may be very altruistic in conception, by the fact that it tends to reward the original inventor, whether he discloses his invention or not, but the application of the system is right away complicated with all the acrobatics of lawyers and all the endless expense it involves. First of all, it is very difficult, in many cases, to determine who is the first inventor.

The German system, not only has the merit of being incomparably simpler, but it rewards the inventor who confers a benefit on the nation by disclosing promptly his invention. In this way, at least, it is more beneficial to the nation at large.

Dr. Ittner also referred to the so-called "claim dodger." The "claim dodger" is a dangerous animal in American patent law. He is one of the worst modern, industrial pirates, although sometimes he sails under the flag of a patentee. His existence in this country is rendered possible by the tendency of Anglo-Saxon law to sacrifice the spirit of any legal document to the letter of it. The German law does not adhere slavishly to the mere wording of the claims of a patent. It takes more in consideration the real substance of the patent. The standpoint of the German patent law is summed up as follows: What was the status of the art, what were the technical efforts possible before the inventor filed his patent, and what advance in the art has been rendered possible after the disclosures contained in the patent?

Just on this account, it is not necessary for a German to have the absurd multiplication of claims which is the characteristic of United States patents, and where quite often the mere cunning use of the English language plays a greater rôle than the invention itself.

NOTES ON A STUDY OF THE TEMPERATURE GRADIENTS OF SETTING PORTLAND CEMENT

By ALLERTON S. CUSHMAN, The Institute of Industrial Research, Washington, D. C.

Read at Joint Meeting with the Eighth International Congress of Applied Chemistry, New York City, September 4-13, 1912.

The reactions that take place when hydraulic cements are tempered with water and while the mixture is hardening are as yet not understood. It is true that many theories have been advanced in regard to the hardening process or processes, but more data is required before much that now seems inexplicable can be understood.

Since all chemical reactions are accompanied by definite and measurable thermal changes, complete temperature records of hardening cements should yield interesting and valuable data.

It is well known that if a Portland cement clinker is ground without the addition of from 2 to 3 per cent of calcic sulphate or gypsum to act as a restrainer it will be "flashy." By "flashy" is meant the tendency to harden very quickly, so quickly in fact that in many cases it is impossible to mold the wetted cement into a plastic mass. While this sudden hardening is going on, a considerable amount of heat is generated so that the mass feels hot to the hand. The temperature rises about 10° to 15° C., but the heat reaction lasts only a short time and after cooling no further heat reaction takes place. When, however, a Portland cement has been properly restrained by grinding with it 2 or 3 per cent of gypsum (plaster) the conditions of thermal activity are changed in a quite extraordinary manner. On mixing a normal Portland cement with sufficient water to form a normally plastic mass, a certain amount of heat is immediately disengaged, although not so much as in the case of an unplastered cement. The plastic mass soon cools down to the air temperature and generally falls somewhat below the surrounding temperature, showing that a decided cooling effect is taking place.

If now the plastic mass is allowed to stand quiescent in a constant temperature chamber, nothing of moment happens if the cement be a normal standard brand, for a period of from four to eight hours. At a given time, however, for every mixture a secondary heat rise begins, and increases more or less rapidly to a definite maximum. After this rise is completed the cement has attained its final set and a gradual cooling takes place to the temperature of the surrounding air and nothing further happens. If an imperfect, damaged or lumpy cement is under observation the temperature gradient for the rise may show aberrations. That is to say, a sudden rise may be followed by a temporary cooling only to be followed by another rise.

The wonderful effect of a small percentage of gypsum plaster in thus controlling and regulating the temperature gradients or reaction of setting cement is little understood and indeed presents certain anomalous occurrences for our consideration, as will be shown later on.

The first successful attempt to record the temperature gradient of setting cement, as far as the writer has been able to ascertain, was made by Gary, who used a photographic recording device, which has been fully described by Burcartz.[1] The method consisted of placing the bulb of an ordinary glass thermometer in the cement paste. The whole arrangement was enclosed in a box through which a beam of light was made to impinge through a slot, upon the graduated stem of the thermometer and then upon a traveling photographic film. As the mercury rose or fell, the beam of light was cut by the shadow of the mercury column and thus a continuous temperature gradient record was obtained.

The only criticism of this method that can be made is that it calls for an expensive and delicately adjusted piece of apparatus which few laboratories would care to install, and in which the temperature changes cannot be watched while they are taking place. The apparatus used by the writer is simple, comparatively inexpensive, and can be installed and used in any laboratory for making daily records. The apparatus is shown in Fig. 1.

A double walled wooden box, as shown in Fig. 1, used simply to avoid any sudden changes which may take place in the laboratory temperature during a test run. An ordinary so-called "fireless cooker," such as can be bought at any kitchen supply store, answers

[1] Eng. Record, Dec. 11th, 1909.

very well for this purpose. The recording thermometer is of the Tycos type and consists of a copper plated steel tapered mercury filled bulb 9 cm. long by about 2 cm. in its maximum diameter. The bulb is connected to the recording dial by a flexible steel capillary tube. The recording dial has a range from 10° to 120° F. The recorder is fairly accurate for the middle range and is easily calibrated and adjusted.

In ordinary tests, as carried out in the writer's laboratory, 1 kilogram of the neat cement is tempered with 250 cc. of water to make a homogeneous plastic paste, which is packed into a No. 2

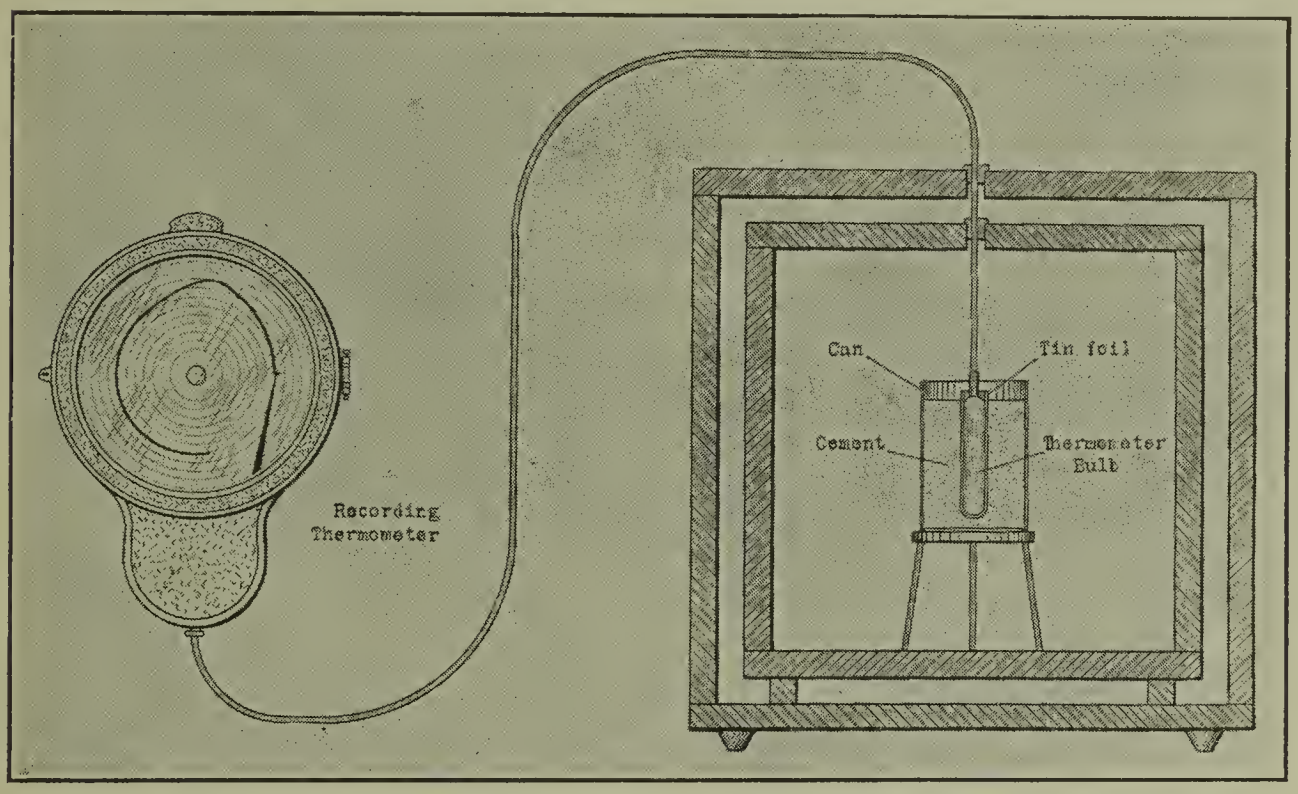
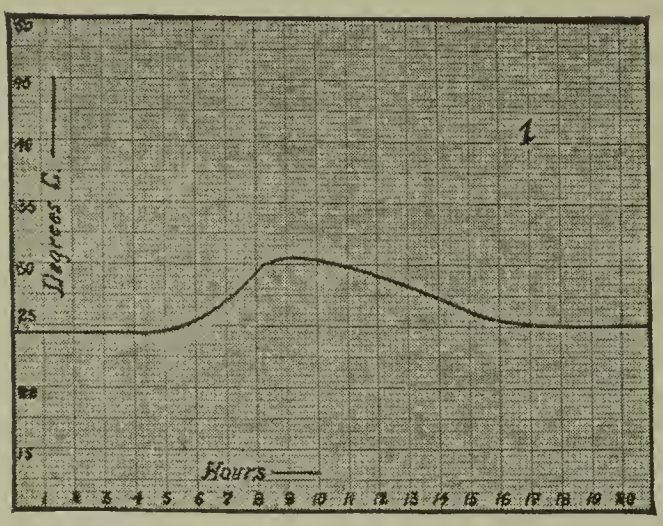
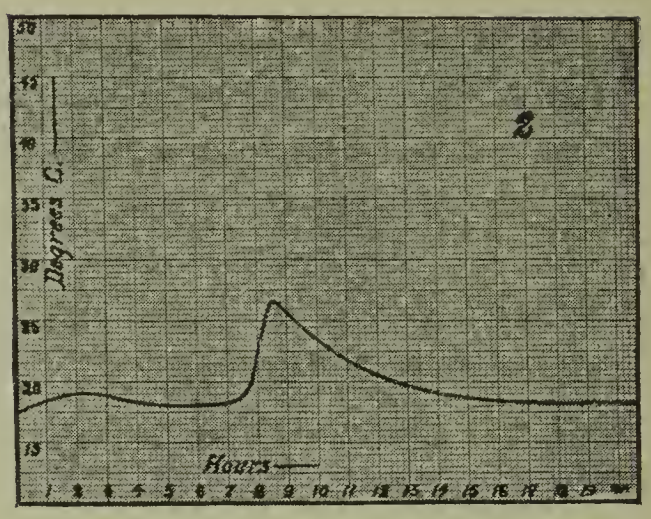
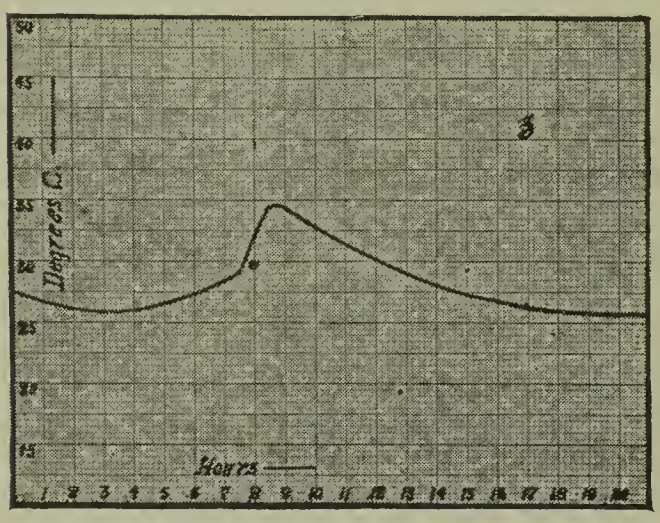


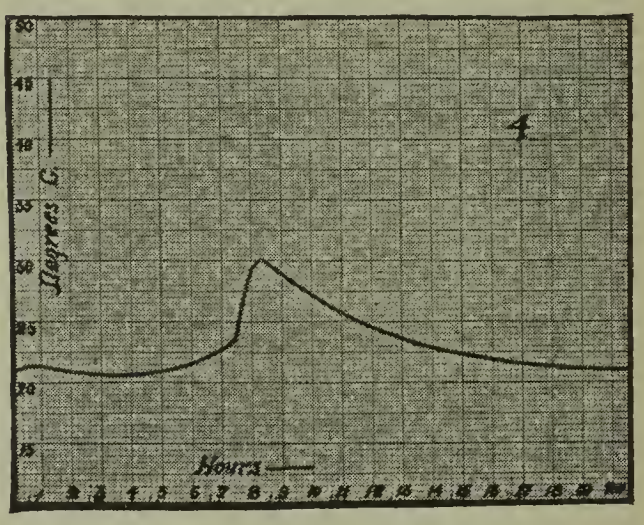
FIG. 1.

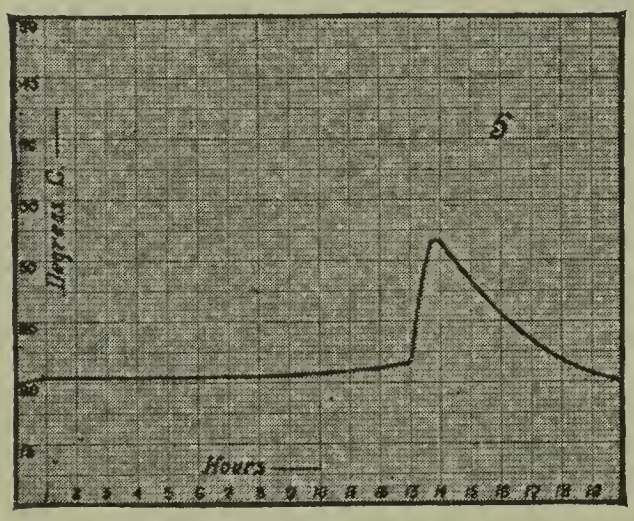
open-top tin can. The thermometer bulb is not inserted until the primary heat effect which always develops when cement is kneaded with water, is over, and the paste reaches approximately the same temperature as the calorimeter box. In the meantime the copper plated thermometer bulb is smeared with vaseline and wrapped with several folds of fairly heavy tin foil. The object of these precautions is two-fold: in part to guard against the "freezing" in of the bulb when the cement hardens and in part to overcome any possible pressure on the walls of the bulb if the cement shrinks or expands during the hardening process. With 25 per cent of water the consistency is somewhat softer than the normal, but experience has

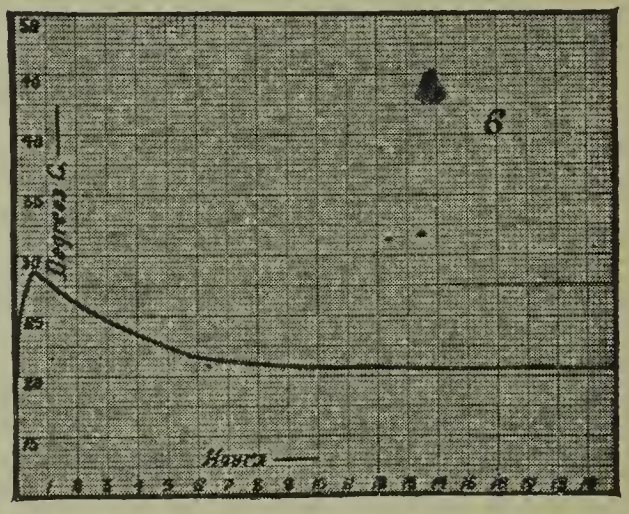

1
Degrees C.
Hours

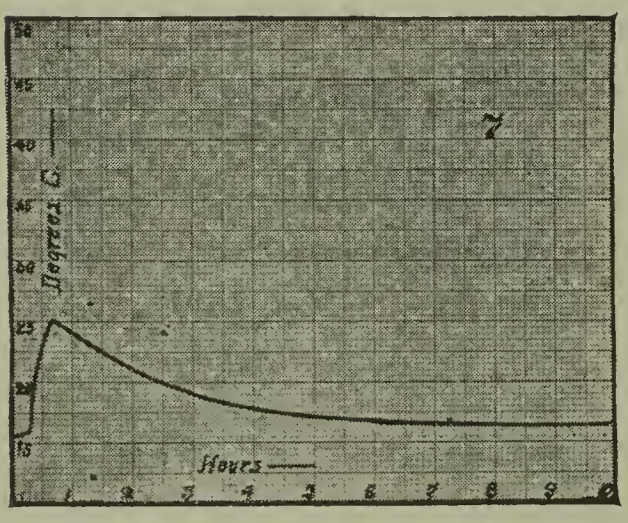

2
Degrees C.
Hours


3
Degrees C.
Hours


4
Degrees C.
Hours


5
Degrees C.
Hours


6
Degrees C.
Hours


7
Degrees C.
Hours

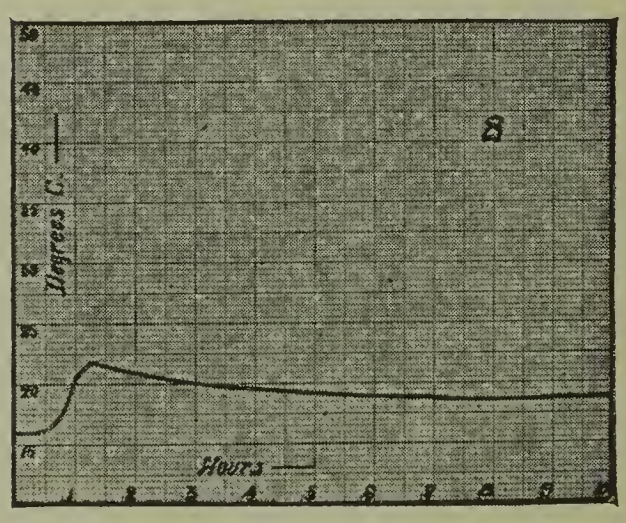
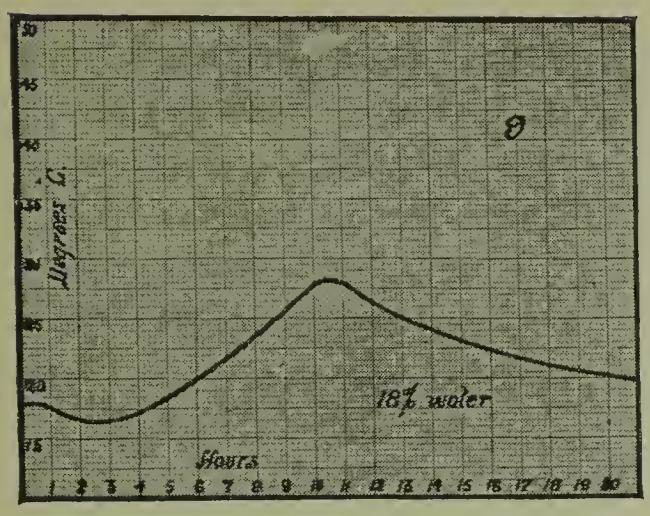
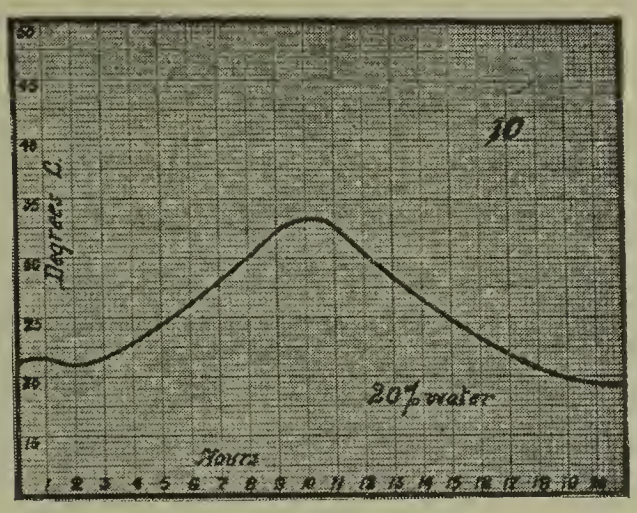
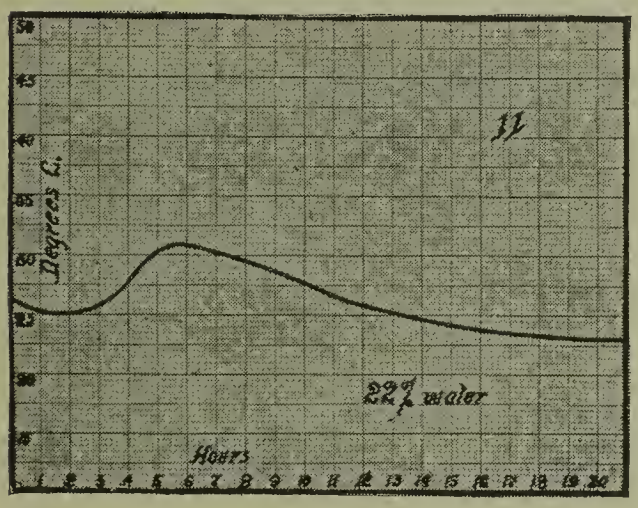
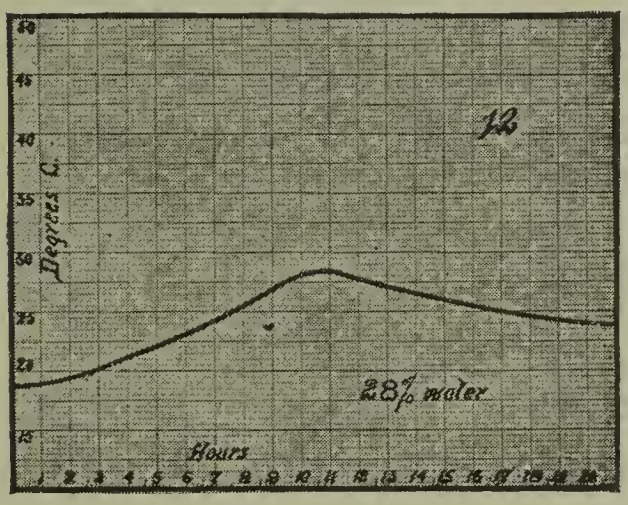

8
Degrees C.
Hours

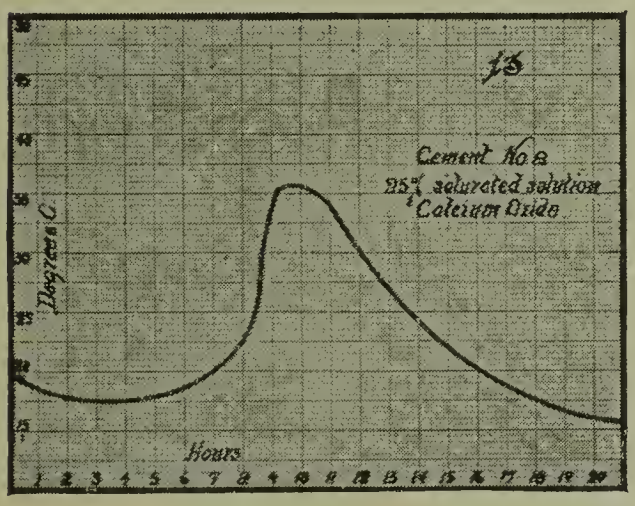
PLATE I.

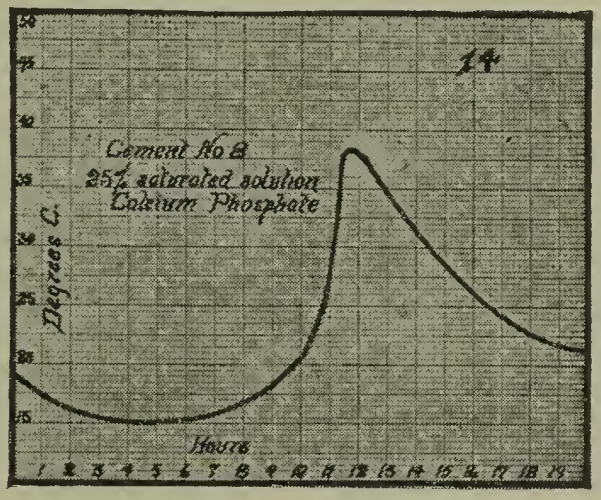

9
Degrees C.
18% water
Hours

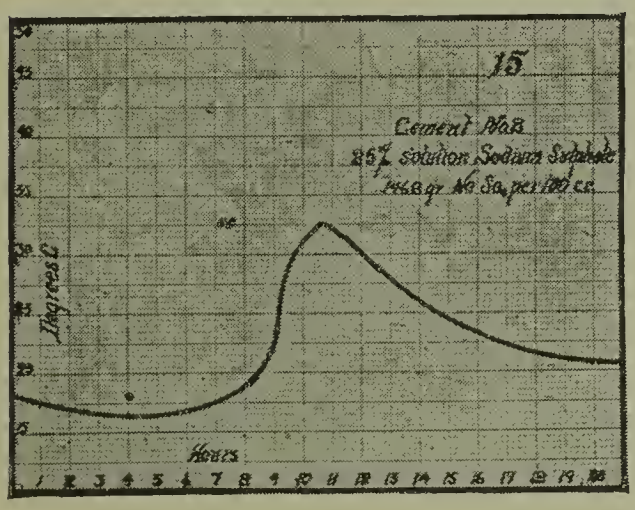

10
Degrees C.
20% water
Hours


11
Degrees C.
22% water
Hours


12
Degrees C.
28% water
Hours


13
Cement No 8
25% saturated solution
Calcium Oxide
Degrees C.
Hours


14
Cement No 8
25% saturated solution
Calcium Phosphate
Degrees C.
Hours


15
Cement No 8
25% solution Sodium Sulphate
1468 gr No So4 per 100 cc
Degrees C.
Hours

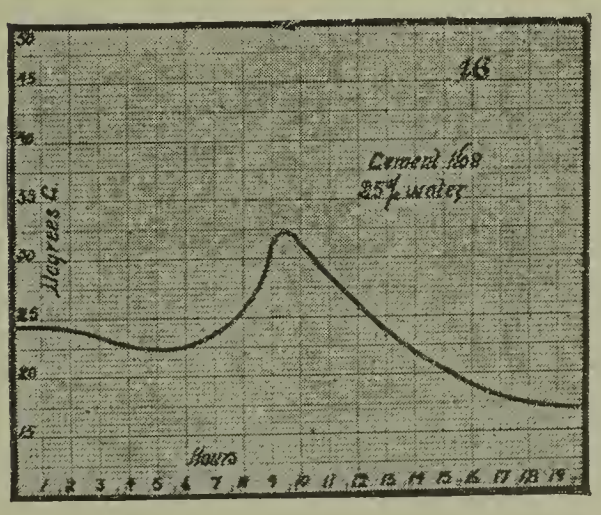
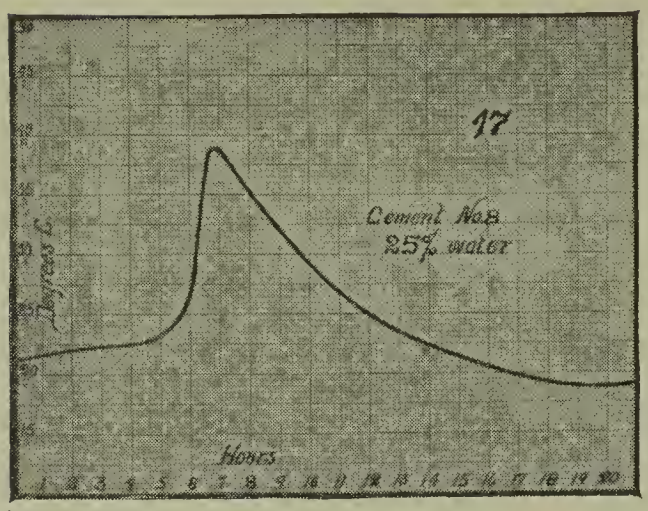
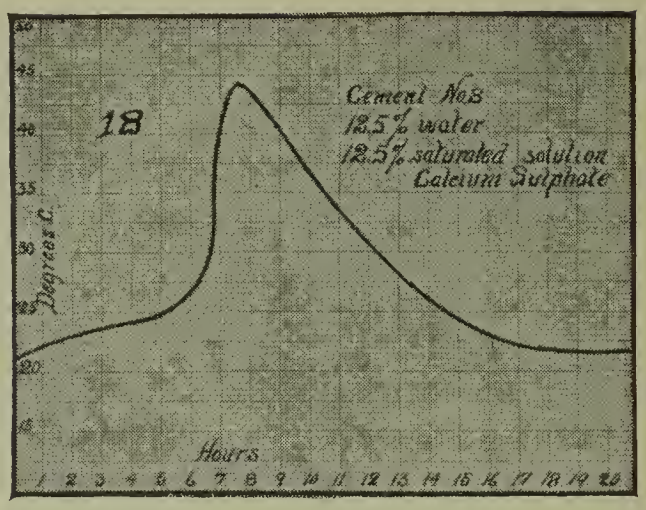
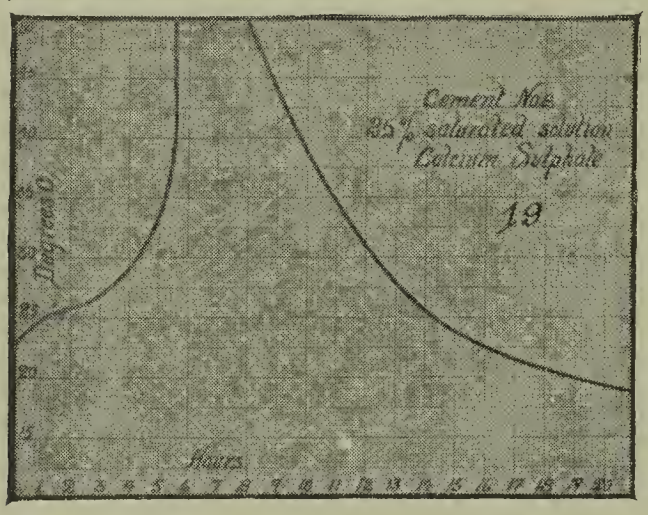
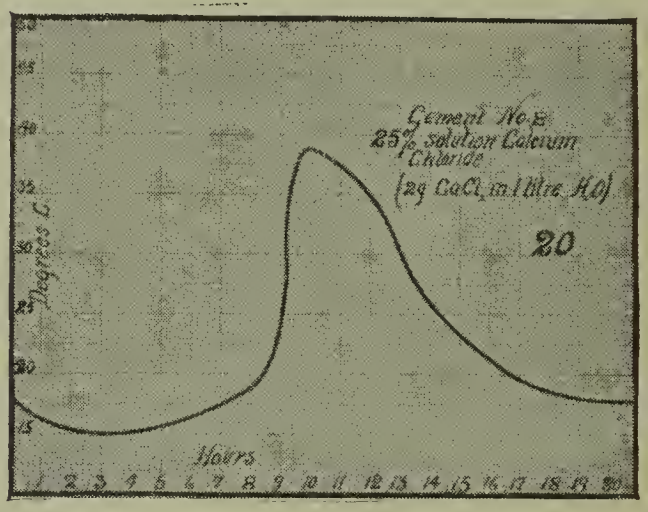

16
Cement No 8
25% water
Degrees C.
Hours

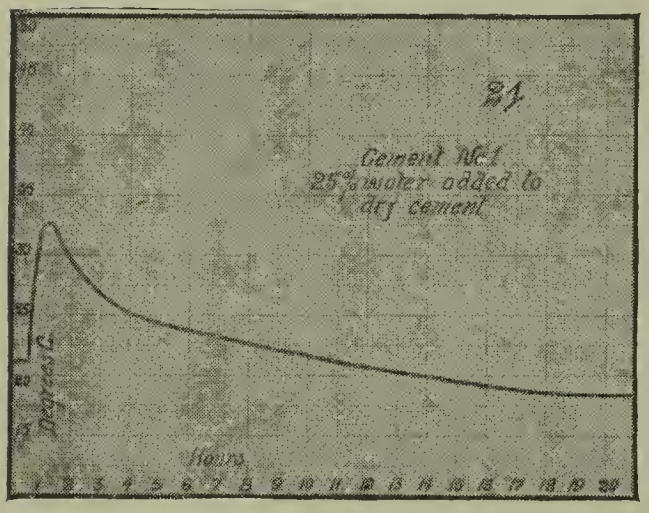
PLATE II.

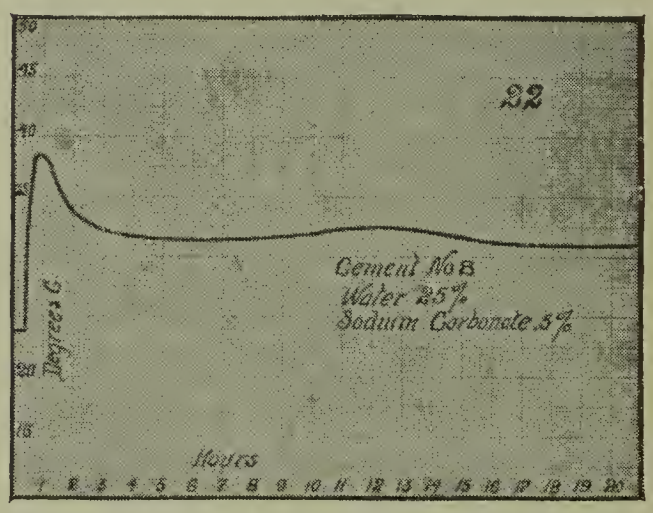

17
Cement No B
25% water
Degrees C.
Hours

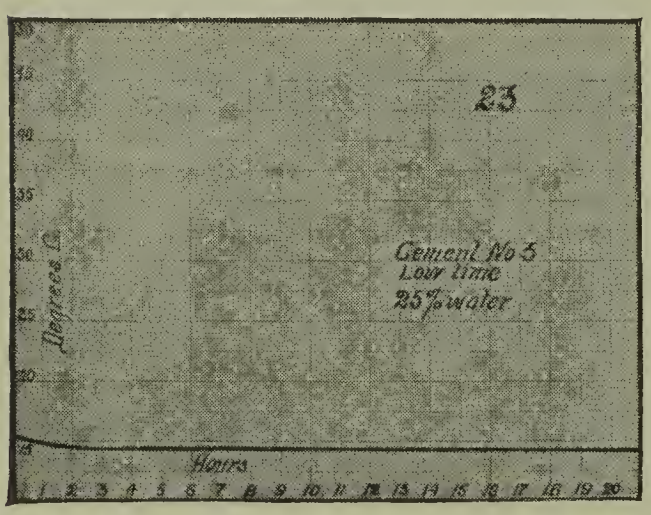

18
Cement No B
12.5% water
12.5% saturated solution
Calcium Sulphate
Degrees C.
Hours


Cement No B
25% saturated solution
Calcium Sulphate
19
Degrees C.
Hours


Cement No B
25% solution Calcium
Chloride
(29 CaCl2 in 1 litre H2O)
20
Degrees C.
Hours


21
Cement No 1
25% water added to
dry cement
Degrees C.
Hours


22
Cement No B
Water 25%
Sodium Carbonate 3%
Degrees C.
Hours


23
Cement No 3
Low time
25% water
Degrees C.
Hours

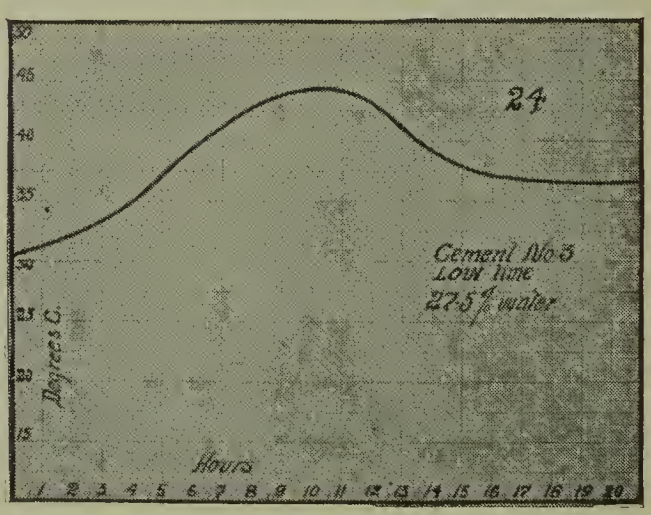
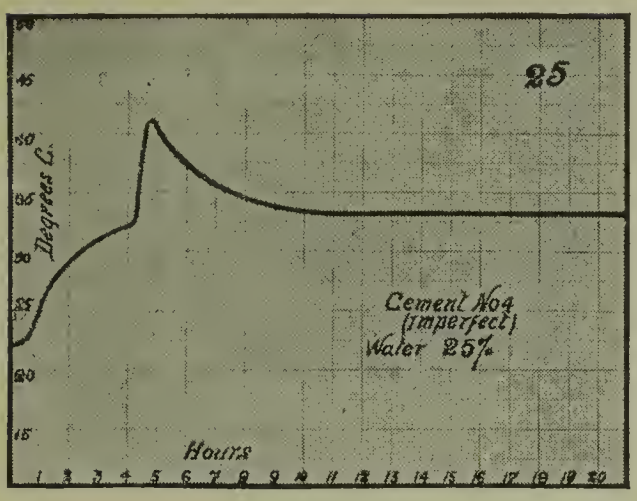
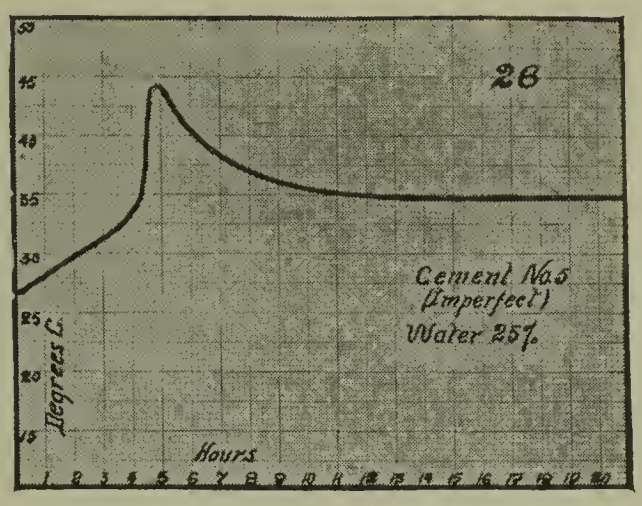
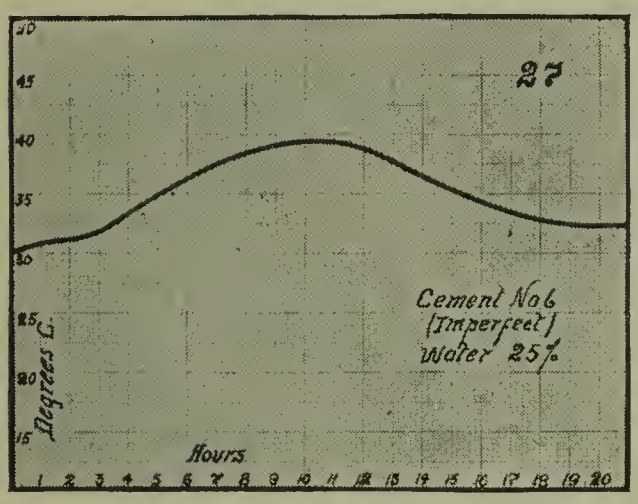
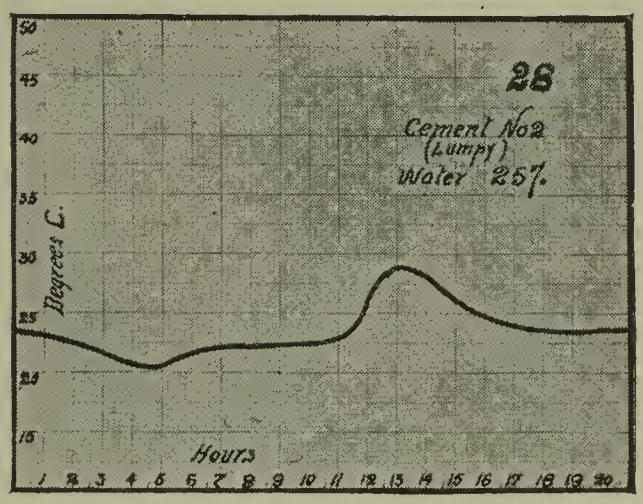

24
Cement No 3
Low time
27.5% water
Degrees C.
Hours

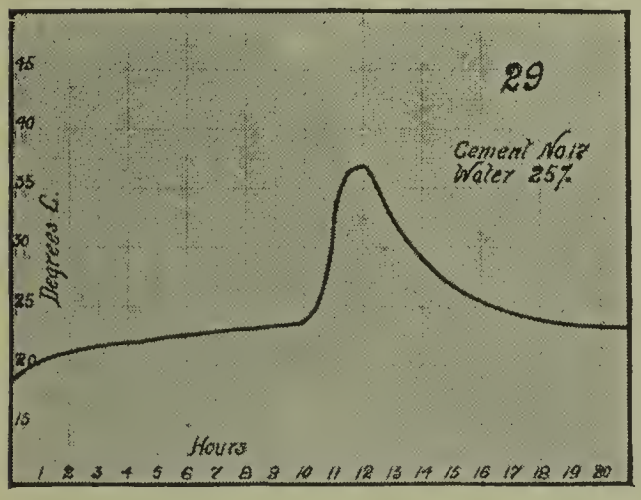
PLATE III.

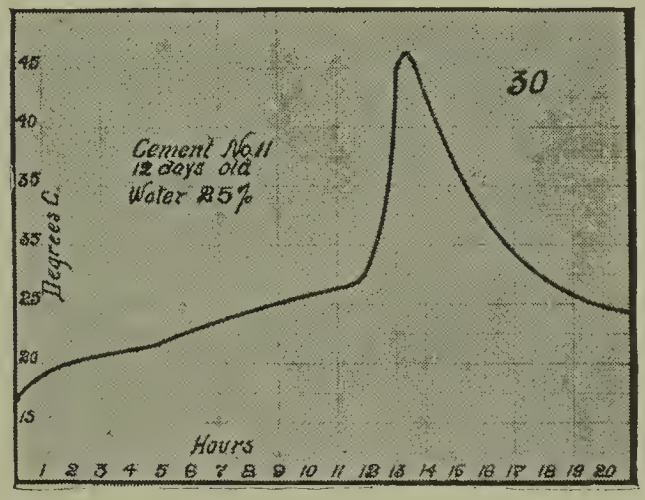

25
Degrees C.
Cement No4
(Imperfect)
Water 25%
Hours

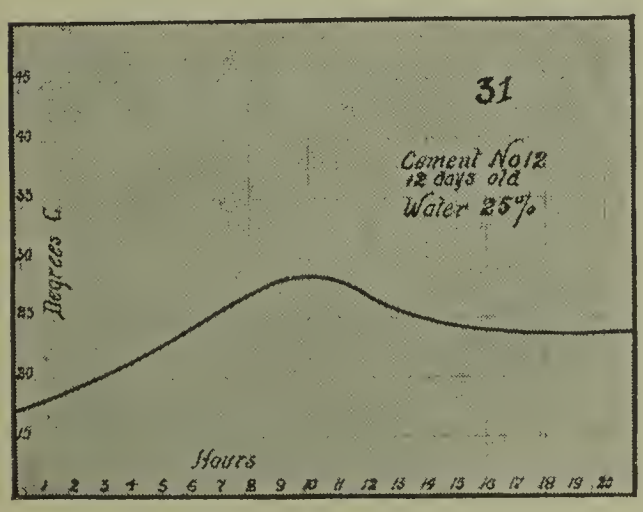

26
Degrees C.
Cement No.5
(Imperfect)
Water 25%.
Hours


27
Degrees C.
Cement No.6
(Imperfect)
Water 25%
Hours


28
Cement No2
(Lumpy)
Water 25%.
Degrees C.
Hours


29
Cement No12
Water 25%
Degrees C.
Hours


30
Cement No.11
12 days old
Water 25%
Degrees C.
Hours


31
Cement No12
12 days old
Water 25%
Degrees C.
Hours

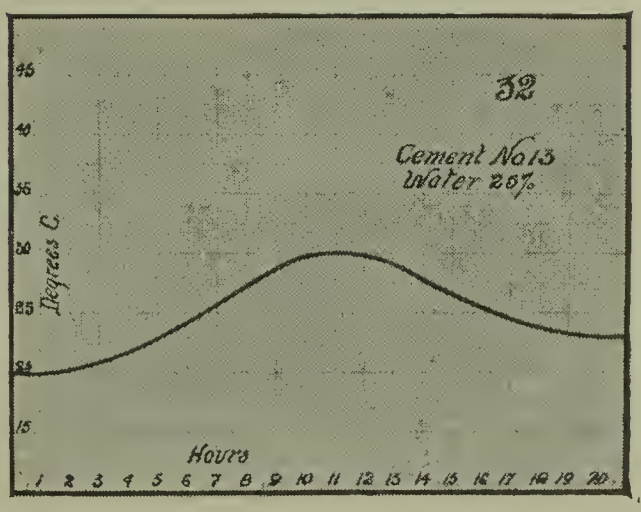

32
Cement No13
Water 26%
Degrees C.
Hours

PLATE IV.

shown that the wetter mixture gives better results under the conditions of the test. When all is ready the covered bulb is pushed into the cement paste, care being taken that it is not pushed below the surface so that the cement can close over the shoulder of the bulb and so imprison it when hardening takes place. When these precautions are taken the apparatus gives no trouble and the bulb is easily withdrawn from the hard cement at the end of the test. The temperature gradients are usually taken for a twenty-four-hour period, although this is not necessary unless the full cooling curve is desired.

In presenting these notes on the temperature gradients of setting cements, it is not the intention of the writer to draw any conclusions at this time in regard to the mechanism of the hardening reactions.

The curves obtained on the revolving scale are transferred to centigrade degrees and plotted in rectangular coordinates as is shown in the illustrations, Plates I, II, III and IV, curves 1 to 32. An inspection of the curves will show that in some cases the temperature gradients are much steeper and more sudden than in others. Curves 6, 7, 8 and 21 represent cases in which the water was simply poured on to the dry cement without previously kneading the mass to a paste. In no case of this kind is a rise in temperature noted following the final set or hardening, at about seven to eight hours.

In the cases of some brands of cement, as is shown in curves 9, 10, 11 and 12, the rise in temperature is constant and gradual to a maximum which usually occurs at about ten to eleven hours. In other cases, notably in curves 17 and 29, the rise is sharp and sudden. As both types of cement pass muster in the standard tests, it is not possible at the present time to state what the ideal temperature gradient curve for a cement should be.

That the maxima and shape of the curves is modified by the addition of various salts to the tempering water, is shown in curves 13, 14, 15, 16, 17, 18, 19, 20 and 22.

Perhaps the most extraordinary curve is number 19, which shows the heating effect produced by saturating the water with calcium sulphate. In this case the temperature rose above the scale of the recording device and the test piece became uncomfortably hot to the hand. Since calcic sulphate is used as a restrainer when

ground with a cement, this extraordinary effect of calcic sulphate solution is difficult to explain.

Curves 25, 26, 27 and 28 were from cements which did not stand test and had been rejected. The abnormality of these curves is at once apparent to the eye and furnishes the best argument as to the value of a study of the temperature gradient as an additional method of control in cement testing.

In conclusion, the author wishes to point out that these notes on the study of temperature gradients are offered not as data on which to establish theories but to stimulate other workers to include similar investigations in their studies of the hardening of hydraulic cements.

THE PRODUCTION OF AVAILABLE POTASH FROM THE NATURAL SILICATES

By ALLERTON S. CUSHMAN and GEORGE W. COGGESHALL,
Washington, D. C.

Read at Joint Meeting with the Eighth International Congress of Applied Chemists, New York City, September 4–13, 1912.

The great demand which has recently arisen for an American supply of potash in available form for agriculture, has stimulated not only the search for new sources of this material, but also experiments on a large and practical scale of operation, in the attempt to develop a method of making the vast supply of potash locked up in feldspars and feldspathic rocks either directly water soluble or sufficiently soluble in dilute acids to insure a product which shall be useful as a fertilizer. The natural silicates commercially available as sources of potash are chiefly orthoclase and leucite. Both of these minerals are potassium-aluminum silicates. The theoretical formula for orthoclase is written $K_2O.Al_2O_3.6SiO_2$, and for leucite $K_2O.Al_2O_3.4SiO_2$. The principal sodium feldspar, albite, has the theoretical formula: $Na_2O.Al_2O_3.6SiO_2$. It is well known that these feldspars run into and substitute each other in various proportions, so that the products from different quarries will vary widely in respect to their soda and potash contents. There is an enormous supply of feldspar in the United States, both east and west, which could be made economically possible as a source of potash, provided the cost of production can be made low enough to compete with the potash-holding manure salts which are at present so largely imported from Germany. Although it must be admitted that the imported potash salts are richer in potash than any product that can ever be made from American feldspars, it should also be remembered that the crude German manure salts contain large quantities of chloride and sulphates of elements which are not only undesirable in the fertilizer but which may do actual harm under certain conditions.

It is this fact which gives encouragement to the attempt to produce from American feldspars a straight potash fertilizer which could be used in exactly the same way that hardwood ashes have been found useful.

Six general methods have been proposed for decomposing the natural silicates in the effort to obtain water-soluble potash salts.

I. Adaptation of Natural Agencies. In the processes of Nature, the slow action of moisture and atmospheric agencies, including the action of carbonic acid gas, is known to have a decomposing or kaolinizing action upon the feldspars. Immense deposits of feldspar and granitic rocks have thus been decomposed, with the formation of large beds of kaolin and clays from which the potash has been leached into the surrounding valley. For this reason, the valleys between feldspathic and granitic hills are usually highly productive of the crops which require large amounts of potash, such as tobacco, potatoes, large fruits, berries, etc. There have been a few processes proposed, which depend principally upon the natural reactions hastened by pressure and other agencies. In 1904 Blackmore (U. S. Patent 772,206) proposed the action of carbon dioxide gas under five hundred pounds pressure upon a cream of the ground mineral, repeated intermittently for several hours, in the attempt to produce a yield of carbonate of potash. Ten years earlier the same experimenter (U. S. patent 513,001) had proposed using lime, calcium chloride and steam pressure in an autoclave to produce chloride. In 1910 Coates (U. S. patent 947,795) proposed the addition of bacteria for the decomposition of feldspar. In 1910 Carpenter (U. S. patent 959,841) proposed to heat the mineral intensely and cool suddenly by plunging in water, in the effort to render the feldspar amorphous, in the hope of making it more available for plant growth. None of the above processes have as yet been shown to possess industrial possibilities.

II. Wet Processes of a Chemical Nature. Levi in 1904 (French patent 344,246 and English patent 13,875) and Piva in 1905 (French patent 351,338) proposed methods for treating leucite by means of solutions of alkali or alkali earth hydrates, generally under increased pressure. The same general method for treating feldspar was claimed by Swayze in 1907 (U. S. patent 862,676) and by Gibbs in 1909 (U. S. patent 910,662).

Also Gibbs in 1904 (U. S. patent 772,612 and 772,657) proposed

a process of treatment with hydrofluosilicic acid, and subsequently with sulphuric acid, in order to produce potassium sulphate. In 1907 Cushman was granted U. S. patent 851,922, a public patent which proposed a sludging of finely ground feldspar with water, the addition of a small amount of hydrofluoric acid and electrolyzing the mixture in wooden cells provided with wooden diaphragms. Under this process both potassium and aluminum hydrate passed through the cell diaphragm into the cathode compartment. This process, although perfectly practical, has not yet been made commercially possible owing to the high cost of hydrofluoric acid and the large amount of by-products formed in the process. None of the above processes have as yet been made commercial possibilities.

III. Dry Processes of a Chemical Nature, in which the Potash Salts are Volatilized. In processes of this nature, fluxes, and in some cases fuel, for producing purposes are ground and mixed with the feldspar, the mixture being subsequently heated until the potash salts are volatilized and collected either in the stack dust or partially collected from the gases by passing them through or over water. Swayze in 1905 (U. S. patent 789,074) heated ground feldspar with gypsum and carbon, and proposed to collect the volatilized sulphate. Spencer and Eckel in 1909 (U. S. patent 912,266) made a cement mixed with calcareous and silicious fluxes and green sand, a natural potash-bearing iron silicate, clinkered the mixture in a rotary cement furnace, and obtained a Portland cement, at the same time collecting the potash in the stack dust and the flue gases. In 1911 Eckel (U. S. patent 1,011,172) proposed a somewhat similar method, but heated only high enough to drive off the potash salts and not high enough to clinker the mixture. Again in 1911 Eckel (U. S. patent 1,011,173) melted a mixture of green sand, limestone and fuel, tapped off the melted iron and slag, and recovered the potash salts from the flue gases.

Some of the processes under this heading have been tried on a large scale. No great difficulty is recorded in driving off the potash in the furnaces, but obstacles were encountered in the attempt to collect the potash from the gases. As a by-product operation in the manufacture of cements, these processes may yet come to be of some industrial importance.

IV. Dry Processes which Propose to Separate Potash as Hydroxide or Carbonate. The old method of Bickell, proposed in

1856 (U. S. patent 16,111), which depended upon heating a mixture of feldspar, lime, and natural phosphate rock or guano to a bright red heat, has not as yet been proved practical or successful. The process of the Soc. Romana Solfati in 1905 (French patent 352,275), which proposes the roasting of leucite with carbonate, hydrate or nitrate of soda and lime and subsequently the passage of steam through the roasted product to produce sodium aluminate and potassium carbonate, is possible from a chemical standpoint, but the high cost of operation has not permitted the process to come into commercial use.

V. Dry Processes Producing the Chloride. These processes have been most experimented with upon the mill scale of operation.

In 1900 Rhodin (U. S. patent 641,406) and in 1901 (J. Soc. Chem. Indus. xx, 439) proposed fritting feldspar with lime and salt. According to the published results, this experimenter obtained good yields although the process has not become a commercial success. In 1907 McKee (U. S. patent 869,011) suggested heating a potash-bearing material containing mica with lime, salt and carbon in order to obtain a yield of potassium chloride. Cushman in 1911 (U. S. patent 987,436) proposed mixing feldspar with lime and salts of a mineral acid capable of decomposing the silicate, giving the mixture special treatment previous to heating in a rotary furnace in order to produce the chloride. This method has been tried out on a large mill scale of operation, and the results obtained will be discussed later on in this paper.

VI. Dry Processes Producing Sulphates. In 1911 Thompson (U. S. patent 995,105) proposed heating to a bright red heat a mixture of feldspar, sodium acid sulphate and sodium chloride, and subsequently leaching out the potassium sulphate produced. This experimenter claims that potassium chloride is first formed, which is subsequently changed to the sulphate by the action of the acid sulphate. It is stated that this process has recently been tried on a commercial scale of operation. Sodium acid sulphate is a by-product that is reasonably cheap, although a large quantity is not available. The lack of an abundant supply of acid sulphite is perhaps the greatest drawback to the commercializing on a large scale of this process, although it is possible that it may still become of some industrial importance. Hart in 1911 (U. S. patent 997,671) proposed to fuse feldspar with some barium compound, such as

the sulphate, together with carbon, to pulverize the cool melt and subsequently to digest the product with sulphuric acid and thus produce in solution potash alum and a residue of barium sulphate and silica, which is claimed to be useful as a paint pigment. Hart claims that some of the potash is volatilized during fusion. Since the fusion temperature is 1500° C., it is probable that a considerable portion of the potash does volatilize, and it is possible that this difficulty may interfere with the commercial success of the process.

Wadman in 1907 (U. S. patent 847,856) proposed heating lepidolite with potassium sulphate and leaching the product with sulphuric acid in order to obtain sulphates of lithium and potash.

A chronological list of the patents which have been granted for the treatment of the silicates for the production of available potash is given in Table I.

It would appear that the most promising processes for making potash available from the natural silicates on a commercial scale of operation are those which are conducted in the dry way but without actual fusion of the reacting mixture. Potash salts volatilize readily at the high temperatures necessary for the fusion of the silicates, and the collection of the volatilized potash from the stack gases has not yet been carried out economically. A considerable portion of the potash does not settle in the dust chamber, and if water sprays are used for washing the gases, the potash solutions are very dilute and the cost of evaporation becomes prohibitive. Furthermore, water sprays are found to interfere with the draft regulation, even when the use of fans is resorted to. The maintenance of artificial draft is an expensive and difficult matter, and is very likely to interfere with the proper control of the furnace temperatures. For work on the large scale of mill operation, a continuous process must be used, avoiding fusion and with the regulation of temperature to the exact point at which appreciable quantities of potash do not volatilize. The fluxes and reacting substances must be cheap, available in large quantity, and the yields of water-soluble potash salts must be high. The process which has seemed to us to give the most promise of successful adaptation to commercial ends is that of Cushman (U. S. patent 987,436) coupled with the method of preparation of the materials before furnacing, proposed and developed by Coggeshall (U. S. patent 987,554).

This process has recently been given extensive trials on a large

TABLE I.—PROPOSED EXTRACTION PROCESSES CHRONOLOGICALLY ARRANGED

	Patentee	Year	Process	Product
IV	Bickell	1856	Lime, $Ca_3(PO_4)_2$, red heat	Caustic
I	Blackmore	1894	Lime, powdered $CaCl_2$, H_2O, steam	KCl
V	Rhodin	1900	Lime, salt, heat under melting	KCl
II	Levi (leucite)	1904	$Ca(OH)_2$ or NaOH, pressure 16 atmospheres	K silicate
II	Gibbs	1904	H_2SiF_6 and H_2SO_4	K_2SO_4
I	Blackmore	1904	CO_2 500 lb. pressure repeating	K_2CO_3
II	Piva	1905	(Leucite) KOH, NaOH, steam 25 atmospheres	K silicate K aluminate
IV	Soc. Romana Solfati	1905	(Leucite) alkali, carbon, CaO, red heat	K_2CO_3
III	Swayze	1905	Gypsum and carbon, fuse, volatilize	K_2SO_4
VI	Wadman	1907	Lepidolite, K_2SO_4 H_2SO_4	K_2SO_4
II	Cushman	1907	Water and HFl, electrolysis	KOH
II	Swayze	1907	Heat alone, then KOH solution	K silicate K aluminate
V	McKee	1907	"Containing mica" with CaO, NaCl, and C	KCl
II	Gibbs	1909	$Ca(OH)_2$, steam 150 lbs.	KOH
III	Spencer and Eckel	1909	Green sand cement mix volatilize	K salts
I	Coates	1910	Bacterial action	
I	Carpenter	1910	Intense heat, sudden cooling alone	
V	Cushman	1911	CaO, $CaCl_2$, etc., clumps, red heat	KCl
VI	Thompson	1911	$NaHSO_4$, NaCl, bright red	K_2SO_4
VI	Hart	1911	Ba compound as $BaSO_4$ and C, fuse, H_2SO_4	Alum
III	Eckel	1911	Cement mix but not over 900° C. with green sand volatilize	K_2O K_2SO_4
III	Eckel	1911	Green sand, CaCO and C, melt iron, volatilize	K_2SO_4

scale and interesting results have been obtained. The process consists essentially in powdering 100 parts of potash feldspar rock together with about 20 parts of lime and with or without 10 to 20 parts of rock salt. This powdered mixture is fed to the top of a moving drum about three feet in diameter, in a layer about half an inch deep. As soon as the layer is formed a strong solution of calcium chloride is applied from a series of small tubes. The $CaCl_2$ at once unites with the lime, forming a so-called oxy-chloride cement, and a large portion of the mixed powder is thereby at once

formed into "clumps" or aggregates lying in a bed of surplus powder. As the drum revolves the bed is removed by a scraper to a belt which delivers the mixture to a screen which separates the clumps from the residual powder. The powder is returned by a screw conveyor and elevator to the hopper above the drum again. The clumps are about the size of peas and pass from the screen directly to a rotary kiln similar to those used in burning Portland cement. The kiln is heated by a blast of air and powdered coal in the usual manner.

The clumps pass regularly down through the increasingly heated portions of the rotating kiln and roll out at the end, practically without alteration in size and shape.

A large percentage of the total potash present in the feldspar is converted into potassium chloride during the heat treatment, and very little is volatilized. The dry clumps are of a pale yellow color outside due to the iron in the ash of the bituminous coal used, but they are snow white inside. The clumps are finally ground, producing a pale yellow material containing as much water-soluble K_2O as hardwood ashes, although the potash is in the form of chloride, and the product also contains considerable free lime. Up to the present time no attempt has been made on a large scale to leach out the soluble potash. The ground material is being given field tests as a straight potash fertilizer containing lime.

A Résumé of the Large Scale Experiments. Potash feldspars were obtained from five different localities. Eleven carloads were used in the trials, amounting to a total of 385 tons. Each carload was ground and analyzed separately. The lowest in potash ran 6 per cent K_2O and 3 per cent Na_2O, the highest 11.3 per cent K_2O and 3.1 per cent Na_2O. The bulk of the spar ran 10 per cent potash and 2 per cent soda, and the results given in this paper were obtained on the 10 per cent spar.

The lime was a high calcium quick-lime, running about 90 per cent CaO and 5.6 per cent MgO.

The salt was rock salt from New York State and ran about 98 per cent NaCl.

The calcium chloride was obtained from the Solvay Process Company. It was in the solid form and contained about 75 per cent $CaCl_2$ and 25 per cent water.

All of the above materials are available in very large quantities

and at low cost. The calcium chloride is a by-product in the form of a moderately strong solution, and but a small proportion is concentrated at the present time, as the chief use is for refrigerating purposes. Vast quantities are now run to waste. The solid form was used in these trials merely for convenience.

Many heats were made with mixtures of varying proportions, but the two mixtures used in the work here described were:

Feldspar	100	Feldspar	100
Lime	20	Lime	20
Salt	10	Salt	20

The feldspar, lime and salt were separately crushed in gyratory crushers and rolls, and dried in a rotary drier. In continuous work the proper mixture would be made at this point by continuous weighing machines, but as a number of different mixtures were to be tried, each of the three raw materials was ground separately in Huntington mills and put into bins. This preliminary grinding of the feldspar and salt was to about 65 per cent through a 100-mesh sieve, of the lime about 83 per cent through the 100-mesh. The weight per cubic foot of each powder of the above fineness was then ascertained and measuring boxes were built so that the materials could be separately measured out and run together into a large mixing machine. Almost a ton was thus mixed each time. The mixture was then conveyed to a tube-mill and further ground to a fineness of from 97 per cent to 99.5 per cent through a 100-mesh sieve, and then conveyed to the bin over the clumper and kiln.

The calcium chloride masses were broken up and thrown on a perforted grid in a large tank holding about 48 tons. Water was run in and the chloride dissolved most readily. The solution was run out when about 42 degrees Beaumé into two large sump tanks and brought to a constant strength of about 42 per cent $CaCl_2$. This was then pumped up to an elevated tank and piped from there, through a constant level tank, to the dropper tubes of the clumper placed in a row above the drum. This drum is 15.5 feet long and 3 feet in diameter, and is horizontal. There are 15 valved pipes, each one feeding an adjustable pipe holding 38 short dropping tubes of brass 1-16 inch internal diameter, and set 5-16 inch apart.

The finely-ground mixed powder is taken from the bin by a chute, elevator and screw conveyor and distributed in a long hopper trough over the drum. It is taken from the trough by a roll device and spread evenly on the moving drum at its topmost point. The drum has a surface velocity of about 1.6 inches per second, the layer of powder advancing at this rate.

It was found that by dropping the liquid very rapidly upon the powder, the clumps could be made rapidly enough to give a full feed to the short rotary kiln when only one-third of the trough and droppers and drum is used. A clumper drum 5 feet long produces every hour almost two tons of fresh clumps and considerably over a ton and a half of burned product with the kiln used in these trials. The excess of powder passes through a screen and goes to the same elevator which lifts the original material from the bin. The amount of actual $CaCl_2$ in the fresh lime is regulated to about 20 parts to each 100 parts of feldspar in the mixture. The clumps leave the screen in rounded form and flow directly into the kiln.

The reason for the above procedure will now be explained. In the first place, calcium chloride reacts very efficiently under these conditions with the feldspar by replacing the potassium with calcium, thus forming calcium silicate and potassium chloride. Anhydrous calcium chloride is expensive to produce and it is impracticable to grind it into a mixture on a large scale on account of the rapid absorption of moisture. Even if such a dry mixture could easily be made, its use would present certain disadvantages.

When a reaction between an ore and solid fluxes is produced by heating up to the fusing temperature, the reaction takes place on the surface of the particles alone and only at the points where the ore is in actual contact with the flux particles. Finer grinding will produce a larger surface area and thus a greater number of actual contact points, leading to a larger yield. There is, however, a degree of fineness beyond which it is not wise to go, on account of the cost of extremely fine grinding.

Another factor in the problem is brought out by the following experiments: A batch of ore and the theoretical amount of solid flux were ground together to just pass a 50-mesh sieve. This powder, when subjected to a certain heat treatment, gave a reaction yield of about 35 per cent of the theoretical. The mixture was then ground to just pass a 100-mesh sieve and given the same heat treat-

ment. A reaction yield was obtained of about 65 per cent of the theoretical. The mixture was then ground to pass a 200-mesh sieve and again reheated as before. A smaller yield was obtained than when the material just passed the 100-mesh, although the particles were undoubtedly only half the average diameter with about four times the surface area, and should therefore have had far more points of contact. Upon weighing equal volumes of the 50-mesh, 100-mesh and 200-mesh powders, it was found that the latter contained far less material and it became apparent that the 200-mesh powder consisted for over 54 per cent of its volume simply of voids. Such finely-ground powders are well known to "surge," that is, to show the tendency to flow like water through orifices in a manner resembling fountains. Material ground as fine as this is the cause of much trouble at spout slides and conveyors. Each particle of a material of this extreme fineness is undoubtedly surrounded by a film of air, the actual contact with the surfaces is lessened and friction almost eliminated. When allowed to flow into a bin, such a powder assumes an almost horizontal surface, there is practically no angle of repose. Unquestionably the lessened contact caused the low yields in the finely ground mixtures. Some of the finer material was briquetted and the subsequent heat yield of about 85 per cent of the theoretical. Briquetting is, however, expensive and usually necessitates the addition of a binding agent foreign to the reaction.

As a result of these investigations, the method was developed for aggregating fine powders by dropping a suitable liquid upon an excess of the powder in such a way as to cause a temporary bond to form, thus practically eliminating the air films or voids around the individual particles and permitting actual surface contact. Under these conditions, with the same ore and flux used in the experiments described above, the same heat treatment yielded within 3 per cent of the theoretical quantity present. This method of aggregating finely-powdered materials previous to furnacing has already been used in several different ways. For example, in an ore mixture in which the fluxing material is an alkaline carbonate, such as sodium or potassium, which forms crystalline salts containing water of crystallization, if the carbonate is used in the partially anhydrous condition and ground with the ore water alone dropped upon the mix in the manner described formed at once a crystalline carbonate

which binds the particles of ore and flux into separate clumps, which are hard enough to withstand screening, while the air films are practically eliminated. Using such a mixture and process as this, a practically theoretical yield was obtained, although the flux was used only in the exact molecular proportion called for by the reaction.

By this clumping process a very intimate contact of reaction of surfaces is readily obtained at a low cost. The quantity of flux necessary to complete the reaction is greatly reduced, the duration and temperature of the heat treatment is lessened, and working with rotary kilns dusting and stack losses are almost entirely eliminated. The clumps are beautifully adapted to the feed mechanism of rotary kilns, as they flow easily, do not dust and take the heat more evenly than fine powders. Now that the temperature conditions in rotary kilns can be accurately controlled, it would seem that many chemical and metallurgical reactions which are now performed by intermittent processes and with low yields could be much more economically carried out in continuous rotary kilns, taking advantage of this new method of forming aggregates previous to furnacing.

In the application of this method to the treatment of feldspathic rock, advantage was taken of the fact that a solution of calcium chloride acts upon dehydrated lime to form the oxychloride, which is a strong cementing compound. It was found that the formation of calcium oxy-chloride gave a sufficiently strong bond to enable the aggregates to withstand the operation of screening and the burden in the kiln.

The theoretical quantity of calcium chloride flux required depends upon the total quantity of K_2O and Na_2O present in the mix, as it is evident that the soda must also be liberated in proportion to its content. The feldspar ore used ran 10 per cent K_2O and 2 per cent Na_2O, which required theoretically 15.5 parts of calcic chloride. In all our trials some slight excess of calcium chloride has been used. The strength of the solution and the method of treatment has been such that about 20 parts of actual calcium chloride are present in the fresh clumps to every 100 parts of feldspar. The 20 parts of lime used is for the purpose of forming the aggregates, and this lime remains practically unchanged in the finished product. The presence of lime in a potash fertilizer will be found advantageous to most soils, and it is generally admitted that lime increases the manurial value of a fertilizer. If the object was to

leach out the soluble potash salts from the product, a much smaller amount of lime could be used without interfering with the formation of hard clumps. The salt is added because it has been found to aid the heat reaction, probably mechanically, as will be explained later on. The fresh clumps contain from 16 to 20 per cent of moisture, which is, of course, evaporated in the upper part of the kiln.

The rotary kiln used in these trials was one of the old bottle-shape cement kilns with a total length of slightly over fifty-five feet, the upper twenty feet having a diameter of four feet clear inside the firebrick lining, the lower portion widening out to nearly six feet inside diameter. The pitch was seven-eights of an inch per foot, and the most suitable speed was found to be one revolution in about two and one-half minutes.

The conditions of the heat treatment are very important. The kiln used was too short to yield the best results, and after the preliminary experiments changes were made which caused the material to take about one and one-half hours to pass through the length of the kiln. The temperature of the gases issuing from the upper end of the kiln were read continually with a thermo-couple pyrometer fitted with a 15-foot fire end and temperatures were also taken from time to time at the firing platform. A furnace wall temperature of about 1370° C. is required for efficient burning of powdered bituminous coal. This is, however, much too high a temperature for potash work in a rotary kiln. This difficulty called for careful experimental investigations and adjustments of the heat treatment before the proper yields could be obtained. If a longer kiln had been available, there is every reason to believe that a more efficient use of the heat could have been obtained. The coal used was a fairly high volatile bituminous coal. It was ground to about 94 per cent through a 100-mesh sieve and blown into the furnace under an air pressure of about ten pounds per square inch.

During the progress of the clumps down the kiln the following reactions probably take place. At the entrance to the kiln the water begins to evaporate. As the hotter zone is approached, the temperature rises high enough to melt calcium chloride and salt. Whether the calcium chloride is free to melt is not known to us, as the exact composition of the oxy-chloride compound formed has not

yet been determined. The results of our work seem to prove that the reacting chlorine is more readily evolved from the oxy-chloride compound than it is from calcium chloride alone. The melting of the salt, however, continues the bond of the reacting particles, causing them to thoroughly "wet" each other, and from this point on the attack on the silicate proceeds rapidly. During the heating usually from 1 to 2 per cent of Na_2O is volatilized.

When operating with no salt present, the yield of soluble potassium chloride was 47.5 per cent of that originally present in the feldspar. On adding to the mixture 10 parts of salt to each 100 of spar, a test heat yielded 64 per cent, but of this 9 per cent was lost by volatilization, giving a yield of 55 per cent net in the final product. On adding 20 parts of salt to the mixture, the yield grows to 69.2 per cent with no volatilization, and to 75 per cent under heat conditions which caused a volatilization of 7 per cent, leaving a net yield of 68 per cent of that originally present. In the case of clumps made from a mixture of 100 parts of feldspar containing 10 per cent K_2O and 2 per cent Na_2O, 20 parts of lime, 20 parts of salt and 20 parts of calcium chloride, the theoretical composition if no volatilization loss takes place, is shown compared with the actual results obtained in the following table:

	Theory	Analysis	
Total K_2O	6.25%	5.8%	
Water soluble K_2O	....	4.2	Equals 6.65% KCl
Loss of K_2O	....	0.5	As KCl already formed
Total Na_2O	7.62	7.1	52% made into NaCl
Water soluble Na_2O	6.37	5.1	Showing 1.79% vaporized as NaCl or 26% of that present.

This particular product contained 11.2 per cent of free lime and total lime by analysis 15.5 per cent. There was also in this sample about 5 per cent of free unchanged calcic chloride. The amount of calcic chloride in the various runs made up to the present time have been reduced gradually to about 1 per cent, and it is felt that in the future better conditions of heat treatment will make complete use of the calcic chloride and at the same time raise the yields of soluble potash. In later runs in which only 10 parts of salt were present in the mix, the theoretical and actual analysis of the product was as follows:

	Theory	Analysis	
Total K_2O	6.66%	5.62%	
Water soluble K_2O	....	4.5	Equals 7.12% KCl
Vaporization loss of soluble K_2O	....	1.04	As KCl already formed
K_2O insoluble in water	....	1.12	
Total Na_2O	4.15		
Water soluble Na_2O	....	3.7	Showing 0.45% vaporized as NaCl or 11% of that present.

This product contained 12.25 per cent of free lime, the total potash rendered soluble was 5.54 per cent of the product or 83.2 per cent of the total quantity present, but as 15.6 per cent had been volatilized the net yield in the product amounted to 67.6 per cent.

The material which was later made continuously according to the process described above carries 4.5 per cent of water soluble K_2O in the form of 7.12 per cent potassium chloride, and in addition to this material carries only 1.12 per cent K_2O insoluble in water. It is well known that a 2 per cent citric acid solution will extract, when used according to the Wagner method somewhat more K_2O than can be made directly water-soluble. This fact is of considerable interest when the product is to be used directly as a potash fertilizer.

Conclusion. It is believed that under better conditions of heat treatment which can be obtained with longer kilns and with a somewhat different arrangement of the combustion chamber slightly better yields than those reported can be obtained. It should be remembered that the kiln used in these experimental trials was originally designed for burning cement, but this type of kiln has long been superseded by improved forms. In order to get the proper heat treatment in the middle of the kiln to complete the reaction, it was necessary to have the upper part too hot. This condition will not maintain in a properly designed kiln. It is also believed that the use of oil as fuel would have allowed an easier regulation of the heat treatment, but the trials so far undertaken have been made under conditions which were found available at the time.

The subject of the costs of this process and of the product cannot be gone into in detail at this time, but a few general statements may be made. The production of water-soluble potash in feldspathic rock is essentially a low-grade proposition, and the commercial success of such a process depends upon the low cost of the various operations. The manufacture of a straight potash fertilizer containing as valuable ingredients only potash and lime must be carried

out on a very large scale and by the most modern methods of continuous operation. With regard to the clumping process, the trials have shown that this operation can be practically carried out as a continuous process and at an exceedingly low charge per ton of product.

The process may be directly compared with that of the manufacture of Portland cement. It is a little easier to grind feldspar and lime than the shales and limestones used in cement manufacture. Drying will cost no more. Chemical control of the raw mixes will not be more expensive and perhaps much less. Clumping, as has been shown, adds a very small charge to the expense of treatment. The cost of furnacing the feldspar mix will be less than similar charges in the cement industry, as the temperatures required are much lower and less coal is consumed. The product from the potash kiln is comparatively soft and pulverizes easily in hammer mills, while the charges on the cement industry for grinding clinker is an important item. Again the softer product merely requires to be ground fine enough for use as a fertilizer, whereas cement clinker must be ground very fine and costs rise rapidly with increasing fineness. Repair bills in the case of feldspar treatment should be much smaller than in cement manufacture. The charge for raw materials is somewhat larger than in the case of cement, but this is more than met by the smaller costs of operation.

The potash fertilizer as now produced should be equal in fertilizing value to the ordinary grades of hardwood ashes. The product carries practically the same content of water-soluble potash and somewhat more lime than wood ashes. There is every reason to believe that if the process becomes an industry the yields of water-soluble potash can be considerably improved. The material yielded is not a fused product, it is friable as an ash and it has the physical texture to make it a valuable aid to soil structure. The success of the product must, of course, depend upon the results obtained under test conditions in its experimental use as a fertilizer. If results are obtained which are as good or better than those which usually attend the proper use of high-grade wood ashes, it is believed that there should be no reason why this product cannot be successfully produced and introduced, especially in those parts of the country where potash feldspars, fuel and shipping facilities are available.

Summary. In this paper a summary is given of the various processes which have been proposed for making the potash in the natural silicates available as a fertilizer.

Experimental trials of a new rotary kiln process for treating feldspar are described, which depends upon a previous treatment before furnacing, consisting of a method of aggregating or clumping the mix so that chemical contact of the reacting substances is brought about during the subsequent processing. The qualitative and quantitative results obtained on a number of experimental trials on a mill scale of operation are presented and discussed. It is shown that it is possible to economically manufacture a potash fertilizer containing free lime from feldspar and for a sufficiently low cost to make an industry based upon the method, worthy of consideration.

POTASH, SILICA AND ALUMINA FROM FELDSPAR

By EDWARD HART

Read at Joint Meeting with the Eighth International Congress of Applied Chemistry, New York City, September 4-13, 1912.

In a study of the commercial utilization of feldspar which I undertook several years ago, it soon became evident that the potash alone would not pay the cost of extraction. This is the cause of the commercial failure of all the methods heretofore proposed. It is necessary, therefore, to separate and put into marketable form the other constitutents—silica and alumina—if our method is to be successful.

With this purpose in view I have finally worked out the following process which gives good prospect of commercial success:

The feldspar chosen should contain not much less than 12 per cent potash. Spar of this quality can be obtained in quantity, but one of the pitfalls inventors must avoid is the expectation of getting spar containing the theoretical 16.9 per cent of potash. The spar mixed with the proper amount of potassium sulphate and carbon is fused. The carbon added is so regulated that the resulting slag contains a considerable proportion of sulphides. This has the double advantage of saving a part of the sulphur, disengaged as hydrogen sulphide on dissolving in acids, which aids also in the complete decomposition by breaking apart the particles as it is given off. Experiments show that if a colorless slag is obtained of even higher alkali content it is much less easily decomposed by sulphuric acid.

The slag so obtained must be very finely pulverized and treated in closed vessels with dilute sulphuric acid leaving behind a very pure silica which needs only washing and ignition to yield a marketable product fitted for the potter's use or for the manufacture of sodium silicate.

The solution contains potash alum and any small amounts of other metals such as iron, manganese and soda as sulphates. Lime is inadmissible, as the sulphate forms crusts on evaporating.

The solution on cooling gives at once crystals of alum, which, washing with a little water and centrifuging, renders marketable. Any iron present remains as ferrous sulphate in the mother liquor. Alum, however, is marketable only in limited quantity and must be, for the most part, converted into its constituents, aluminum and potassium sulphates. This is easily done by adding to the solution in a closed vessel potassium sulphide in slight excess when aluminum hydroxide mixed with a little sulphur precipitates in a form easily washed. This is dissolved in hot sulphuric acid, run through a filter and allowed to solidify. The potassium sulphate is obtained by evaporation.

Each ton of feldspar (12 per cent K_2O) should yield 444 lbs. K_2SO_4, 2040 lbs. commercial aluminum sulphate (18 per cent Al_2O_3), and 1300 lbs. SiO_2.

GAYLEY CHEMICAL AND METALLURGICAL LABORATORY,
LAFAYETTE COLLEGE,
EASTON, PA.

A CHEMICAL INVESTIGATION OF ASIATIC RICE

By ALLERTON S. CUSHMAN and H. C. FULLER, Institute of Industrial Research, Washington, D. C.

Read at Joint Meeting with the Eight International Congress of Applied Chemistry, New York City, September 4-13, 1912.

INTRODUCTION.—The following paper contains a description and the results of a complete chemical investigation of twenty-seven samples of Asiatic rice, which was recently carried out at the instance of the Siamese Government. The samples were collected in the open maket at Singapore and Shanghai and no effort was made to prepare them in any way differently from those rices which are ordinarily exposed for sale in the Asiatic market. The relation of an exclusive rice diet to the etiology of beri-beri disease has been much discussed for a number of years past. This paper does not pretend to decide this controversy but is offered as a contribution to the general knowledge of the chemical constitution of rice. As far as the authors are aware the results on the phosphate content of eastern rices is the most complete yet published.

DESCRIPTION OF SAMPLES.—The samples reached the Institute on October 30th, 1911, and the box containing them was opened on October 31st. The samples were contained in twenty-seven 10 pound cotton bags numbered serially 1 to 27. No other distinguishing marks or information was found.

The cotton bags were found to be frail and rotten and in some cases were broken through, so that the contents had partially escaped. All the samples contained living weevils, and a few worms and beetles were also found. The condition of the samples made it necessary to hand pick them to remove insects. They were then immediately packed in glass bottles, stoppered and labeled.

The appearance of the samples indicated that they represented a medium grade of white or milled rices. On the trip from the Far East the samples had evidently suffered desiccation with the

result that some of the grains had become abraded and broken. As it was not believed, however, that the grain had suffered in such a way as to affect the chemical analysis except in regard to moisture content and the weight per 100 grains, it was decided to be unnecessary to delay the investigation by awaiting a new importation of samples from the Far East.

THE ANALYTICAL WORK.—The analytical work was carried out by the methods recommended by the Association of Official Agricultural Chemists of the United States, and comprised the following elements usually sought: Moisture, Ash, Proteids, Ether Extract (mainly Fat), Fibre, Starch and other Carbohydrates, Weight per 100 Grains.

The above determinations have usually been accounted sufficient to fix the nutrition value of a given cereal. In view, however, of a recently published claim that milled rices are deficient in organically combined phosphorous, phosphate determinations were carried out on each sample. The results have been carefully checked and may be taken as accurate for the samples worked on.

TABULATION OF RESULTS.—The results of the analytical work on the twenty-seven samples submitted are given in Table I, with the exception of the phosphate contents which are tabulated separately in Table III. Table II gives the results of analysis of two fresh samples of South Carolina (U. S. A.) rices bought at a prominent grocery house in Washington, D. C. These samples are denominated Numbers 29 and 30. Sample 29 is the ordinary very white large grained rice as sold in the United States at about ten cents a pound. Sample 30 was sold for a slightly higher price and purported to be a "natural uncoated special pure rice." Table III gives the phosphate content of all samples, reported as phosphoric anhydride, P_2O_5. In Appendix A are given the results of an examination of various rices exhibited at the World's Columbian Exhibition, at Chicago, in 1893, the analyses made by the Division of Chemistry, U. S. Department of Agriculture. Appendix A is preceded by an extract from Bulletin No. 13, and is followed by a summing up of the results.

TABLE I

RESULTS OF ANALYSIS OF TWENTY-SEVEN SAMPLES OF RICE SUBMITTED TO THE INSTITUTE OF INDUSTRIAL RESEARCH BY THE SIAMESE LEGATION, WASHINGTON, D. C.

Sample No.	Weight of 100 Grains.	Moisture.	Ash.	Ether Extract.	Crude Fiber.	Proteids.	Starch and Carbohydrates.
1	1.565 gms.	11.02%	0.46%	0.31%	0.40%	8.13%	79.68%
2	1.539 "	10.99	0.51	0.29	0.60	8.25	79.36
3	1.181 "	11.11	0.56	0.20	0.29	7.38	80.46
4	1.036 "	10.82	0.46	0.15	0.20	8.44	79.93
5	1.708 "	11.54	0.40	0.13	0.82	8.44	78.67
6	1.651 "	10.51	0.49	0.28	0.83	7.56	80.33
7	1.498 "	11.14	0.50	0.20	0.72	7.81	79.63
8	1.244 "	11.31	0.48	0.15	0.47	7.75	79.84
9	1.481 "	11.10	0.55	0.68	0.66	8.31	78.70
10	1.409 "	11.30	0.41	0.63	0.43	7.81	79.42
11	1.329 "	10.60	0.49	0.20	0.21	7.63	80.87
12	1.725 "	11.28	0.47	0.31	0.27	7.56	80.11
13	1.723 "	10.45	0.45	0.17	0.60	8.06	80.23
14	1.541 "	10.94	0.44	0.53	0.76	7.56	79.77
15	1.141 "	10.44	0.54	0.10	0.31	7.81	80.80
16	.....	11.08	0.85	0.28	0.44	8.25	79.10
17	0.958 "	10.51	0.74	0.12	0.16	7.81	80.66
18	0.892 "	10.49	0.60	0.30	0.32	8.00	80.29
19	0.788 "	9.99	0.48	0.94	0.33	8.06	80.20
20	.....	10.06	0.55	0.71	0.51	8.13	80.04
21	1.238 "	9.21	1.23	0.80	0.77	8.44	79.55
22	1.175 "	9.19	0.72	0.87	0.56	8.94	79.72
23	1.533 "	9.32	0.57	0.52	0.45	8.75	80.39
24	1.179 "	9.55	0.77	0.91	0.47	8.38	79.92
25	1.429 "	10.37	0.58	0.16	0.23	8.38	80.28
26	1.413 "	10.04	0.72	0.59	0.45	7.63	80.57
27	1.581 "	10.81	0.51	0.44	0.31	8.63	79.30

TABLE II

RESULT OF ANALYSIS OF TWO SAMPLES OF SOUTH CAROLINA RICE

Sample No.	Weight of 100 Grains.	Moisture.	Ash.	Ether Extract.	Crude Fibre.	Proteids.	Starch and Carbohydrates.
29	2.241 gms.	10.23%	0.47%	0.42%	0.29%	9.00%	79.59%
30	2.238 "	9.01	0.37	0.21	0.36	8.13	81.92

TABLE III

RESULTS OF PHOSPHATE DETERMINATIONS ON TWENTY-SEVEN SAMPLES OF RICE SUBMITTED TO THE INSTITUTE OF INDUSTRIAL RESEARCH BY THE SIAMESE LEGATION, WASHINGTON, D. C.

Sample No.	Per Cent P_2O_5.	Sample No.	Per Cent P_2O_5.	Sample No.	Per Cent P_2O_5.
1	0.22	10	0.31	19	0.31
2	0.39	11	0.32	20	0.30
3	0.30	12	0.23	21	0.41
4	0.20	13	0.21	22	0.39
5	0.28	14	0.21	23	0.42
6	0.26	15	0.30	24	0.58
7	0.31	16	0.49	25	0.24
8	0.26	17	0.35	26	0.22
9	0.30	18	0.35	27	0.34

South Carolina rice.....	29	0.29
	30	0.24

INTERPRETATION OF RESULTS.—A careful inspection of the results shows, that all of the analyses of the samples submitted compare favorably in respect to nutrition value with the samples given under the World's Fair report which includes typical rice analyses as quoted by various authorities (see Appendix A). The results also for the most part compare well with the analyses of the South Carolina rices given in Table II. The phosphorous content of the imported samples (Table III) shows considerable variation; in some cases it corresponds to the average for milled white rice which is reported to be about 0.25%; in other cases it is as high as is usually shown in rices treated by the parboiling process. It would appear that the white rices as represented in the twenty-seven imported samples show on the average as high a nutrition value as the white rices from other sources. The moisture content and weight per 100 grains is somewhat low in the imported samples, for the reason stated above.

INTERPRETATION OF THE ANALYTICAL RESULTS IN RELATION TO THE ETIOLOGY OF BERI-BERI.—It has recently been claimed by Doctors Fraser and Stanton of the Institute for Medical Research, Kuala Lumpor, that the low phosphorous content of white milled rices is a predisposing cause of beri-beri. (See "The Lancet,"

London, Vol. 176, p. 451, 1909.) It is further stated by Doctors Fraser and Stanton that: "From epidemilogical conditions and from experimental evidence it appears that Siam rice is considerably more potent in its beri-beri producing powers than Rangoon rice."

Opposed to the conclusions of Doctors Fraser and Stanton stands the opinion of Dr. Hamilton Wright, former Director of the Institute for Medical Research, Federated Malay States, an eminent investigator of the Etiology and Pathology of Beri-beri. Dr. Wright's published opinion,* based on years of study and clinical experimentation is quoted below:

"The theory of the causation of beri-beri that fits the above facts and all others observed in British Maylaya is that beri-beri is due to a specific organism which gains entrance to the body via the mouth, that it develops and produces a toxin chiefly in the pyloric end of the stomach and duodenum, and that the toxin, being absorbed, acts atrophically on the peripheral terminations of the afferent and efferent neurones. Further, that the specific organism escapes in the fæces and lodges in confined places through accident or the careless personal habits of those affected by the disorder, and that in the presence of congenial meteorological, climatic and artificial conditions of close association from overcrowding, the organism becomes virulent and, gaining entrance to the healthy body in food, etc., contaminated by it, gives rise to an attack of the disease. The fact that the germ remains so closely focal can, I think, be explained by its being at once destroyed by the action of direct sunlight or that the presence of CO_2 or some other gas is necessary for its virile development. It seems from my observations here that the active stage of the organism in the body is between three and four weeks. I base this estimation on the facts that the preliminary feeling of oppression in the epigastrium ceases at the end of about three weeks, and that it is rare to find the lesion of the gastric and intestinal mucose in cases of only six weeks' standing."

Conclusion.—As far as the results of analysis can be interpreted in the light of the information at hand, there would appear

* An inquiry into the Etiology and Pathology of Beri-beri. Hamilton Wright, M. D., Studies from Institute for Medical Research, Federated Malay States, Vol. 2, No. 1, p. 58 (363).

to be no reason why the white milled rices from one section of the world should be held more responsible for mal-nutrition than similar rices from other sections.

APPENDIX A.

Extract from Bulletin No. 13,* U. S. Department of Agriculture, Division of Chemistry.

Rice may reach the analyst in three different states, viz.: unhulled, hulled, and polished. He may also have occasion to examine the broken fragments used in polishing and hulling, the waste in manufacturing rice bran and other products. The most important of these products in the present connection is the polished rice as it is found in commerce, ready for preparation as food. Rice is a cereal in which the starchy matters predominate, and in which there is a marked deficiency of proteids and oils as compared with other standard cereals. The composition of rice, as determined by the analysis of samples exhibited at the World's Columbian Exposition, and by standard authorities, is best shown in the table of maxima, minima, and means, as in the case of the other cereals which have been mentioned. In the following table the items marked I, II, and III, represent data obtained at the World's Columbian Exposition, while the means of all the samples there analyzed are given in another part of the table.

The mean composition of the different classes of rice as shown by the analyses of the World's Fair samples is almost the same as that shown by the work of other analysts collated as indicated above. A typical unhulled rice has about the following composition:

Weight of 100 kernels, grams	3.00	Crude fibre, per cent	9.00
Moisture, per cent	10.50	Ash, per cent	4.00
Proteids, per cent	7.50	Carbohydrates, other than crude fibre, per cent	67.40
Ether extract, per cent	1.60		

*Foods and Food Adulterants. Investigations made under direction of H. W. Wiley, Chief Chemist, Part 9. Cereals and Cereal Products, Washington, D. C., 1898.

TABLE OF MAXIMA, MINIMA, AND MEANS OF CONSTITUENTS OF RICE

Kinds and Numbers of Samples.	Weight of 100 Kernels.	Moisture.	Proteids.	Ether Extract.	Crude Fibre.	Ash.	Carbohydrates, Excluding Fibre.
	Grams.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
1. Rice in the hull (foreign):							
Maxima	3.250*	11.52†	8.40†	2.04†	11.47†	4.66*	65.70*
Minima	2.842†	9.03*	8.23*	1.44*	9.45†	3.26†	65.01*
Means	2.979	9.88	8.32	1.71	10.62	4.12	65.35
2. Unpolished rice (foreign):							
Maxima	2.826‡	12.57‡	10.50‡	2.26‡	1.00‡	1.22‡	77.34‡
Minima	2.260‡	10.92‡	7.27‡	1.62‡	0.87‡	1.04‡	73.35‡
Means	2.466	11.88	8.02	1.96	0.93	1.15	76.05
3. Polished rice (foreign):							
Maxima	2.633†	13.15†	10.33†	0.54‡	0.56*	0.65*	81.66‡
Minima	1.560*	11.82‡	5.42‡	0.04‡	0.27*	0.28*	75.62†
Means	2.132	12.34	7.18	0.26	0.40	0.46	79.36
Mean composition of polished rice, etc., as given by Jenkins and Winton.							
Polished rice (10 analyses)	.....	12.40	7.40	0.40	0.20	0.40	79.20
Rice bran (5 analyses)	.....	9.70	12.10	10.90	9.50	10.00	49.90
Rice hulls (3 analyses)	.....	8.20	3.60	0.70	35.70	13.20	38.60
Rice polished (4 analyses)	.....	10.00	11.70	7.30	6.30	6.70	58.00
Mean composition of rice, etc., as given by Konig.							
Unhulled rice (3 analyses)	.....	11.99	6.48	1.65	6.48	3.33	70.07
Hulled rice (41 analyses)	.....	12.58	6.73	1.88	1.53	0.82	76.46
Polished rice (9 analyses)	.....	12.52	7.52	0.84	0.48	0.64	78.00
Means of World Fair samples.							
Unhulled rice (4 analyses)	2.929	10.28	7.95	1.65	10.42	4.09	65.60
Unpolished rice (6 analyses)	2.466	11.88	8.02	1.96	0.93	1.15	76.05
Polished rice (14 analyses)	2.132	12.34	7.18	0.26	0.40	0.46	79.36

* Guatemala. † Johore. ‡ Japan.

A typical hulled rice, but unpolished, has about the following compositions:

Weight of 100 kernels, grams	2.50	Crude fibre, per cent	1.00
Moisture, per cent	12.00	Ash, per cent	1.00
Proteids, per cent	8.00	Carbohydrates, other than crude fibre, per cent	76.00
Ether extract, per cent	2.00		

A typical polished rice has a composition represented by the following numbers:

Weight of 100 kernels, grams	2.20	Crude fibre, per cent	0.40
Moisture, per cent	12.40	Ash, per cent	0.50
Proteids, per cent	7.50	Carbohydrates, other than crude fibre, per cent	78.80
Ether extract, per cent	0.40		

THE BEEHIVE COKE OVEN INDUSTRY OF THE UNITED STATES

By A. W. BELDEN,* Bureau of Mines Experiment Station, Pittsburgh, Pa.

Read at Joint Meeting with the Eighth International Congress of Applied Chemists, New York, September 4-13, 1912

The manufacture of coke in the United States according to authenticated reports was begun about 1817. From this date on, mention is made from time to time of the use of coke for metallurgical purposes, but the coke was made on the ground in pits or mounds and no record of any coke made in ovens can be found until the year of 1841 when two carpenters and a stone mason formed a partnership for the building of two ovens and the manufacture and sale of coke. This plant was built in the famous Connellsville region, and although the business venture was unsuccessful the coke proved useful for foundry purposes. This venture, together with experiments carried on during the next ten or fifteen years, fully demonstrated the value of this fuel, and production increased by leaps and bounds into the vast beehive coke oven industry which completely dominated the field until 1893, when the first coke oven plant for the recovery of by-products was introduced into this country.

The evolution of the modern beehive oven started as shown above and the process of coking in this type of oven has not materially changed, the modern increased efficiency being due in great measure to improvements in the ovens and the preparation of the coal before charging into the ovens.

BEEHIVE OVENS

The beehive oven in its essential details may be described as a circular vaulted fire-brick chamber constructed on a suitable foundation, with flat tile bottom, an opening in the top through

* Published by permission of the Director of the U. S. Bureau of Mines.

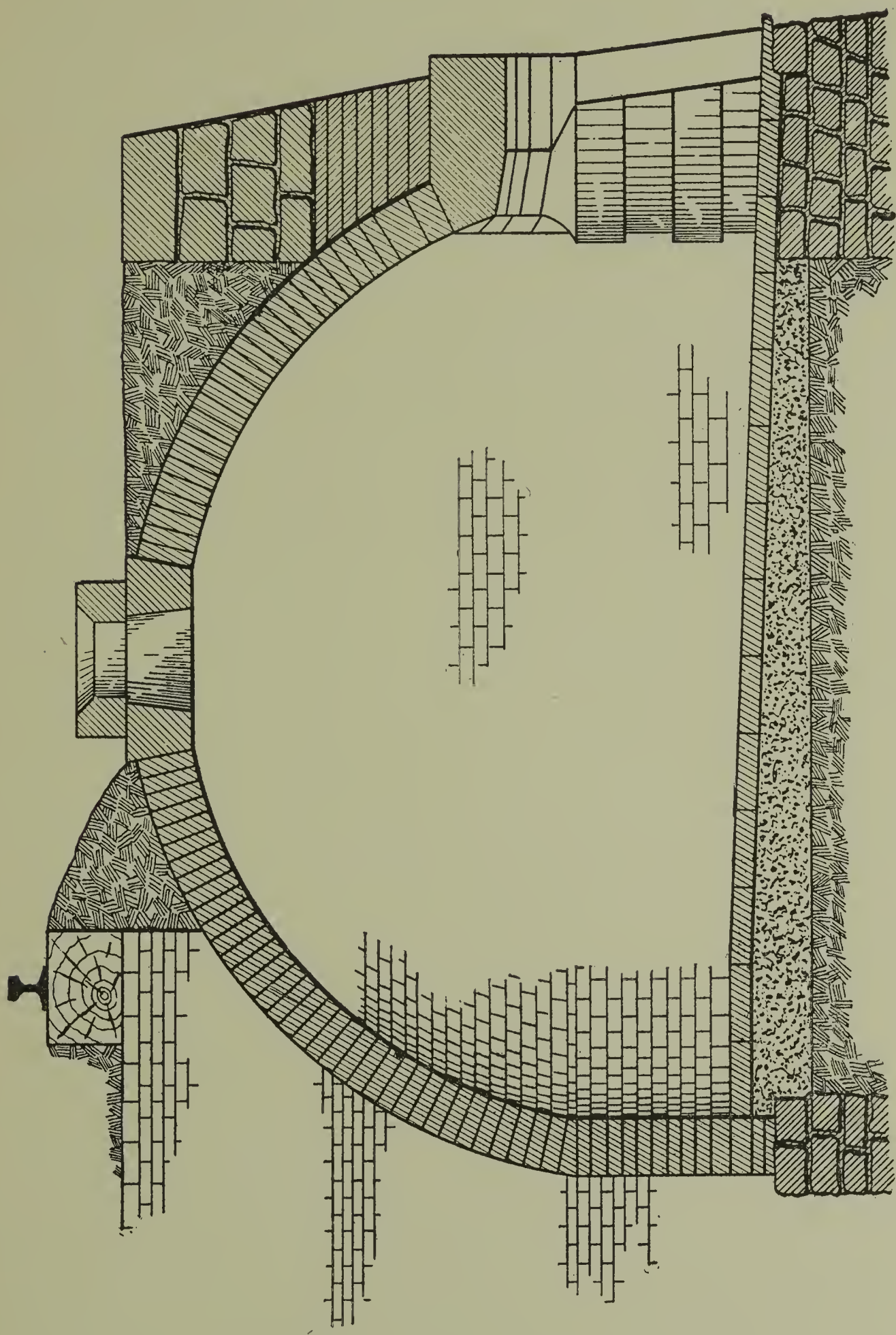


FIGURE 1—BEEHIVE OVEN SECTION OF STANDARD TYPE. 12′ DIAMETER, 7′ 6″ HIGH.

which the coal is charged and the products of combustion escape, and an arched door at the bottom about three feet high, through which the air for combustion is admitted and the coke watered and drawn. The many variations and improvements since the ten-foot-diameter oven of the carpenters and stone mason of 1841 have led to the present standard beehive oven as shown in section Figure 1. The size of the door and the trunnel-head, height of dome and diameter of oven vary in different localities, but the essential features are the same. The ovens are built in single rows called bank ovens or in double rows back to back or staggered. The coal, the amount of which is previously determined as the oven is to be burned 48, 72 or 96 hours, is charged into the oven from a larry car operated on a track above the ovens. The coal in falling into the oven forms a conical pile and is leveled by means of a scraper operated by hand. The door is bricked up to within two or three inches of the top and the oven left until the heat held by the bricks from preliminary heating, or the previous charge, raises the temperature to the point where the volatile matter, distilled from the coal, finally catches fire. The coking proceeds from the top downwards and the burning of the volatile to maintain the required heat is regulated by closing up the air opening as the amount of the volatile falls off. After the volatile has ceased to come off the process is finished. The door is then torn down, the coke watered on the inside of the oven by means of a spray of water and is then drawn from the oven.

The beehive oven arranged for the mechanical drawing of coke differs from the above only in the width of the door and projecting iron jams at the sides of the door. Mechanical drawing of the beehive oven has been resorted to on account of the scarcity of labor and not from any increased efficiency resulting from the mechanical operation *per se*. There is reason to doubt if mechanical drawing shows any material reduction in the cost of operation when all the items are taken into consideration. It undoubtedly breaks up the coke more, producing a large percentage of breeze, but, on the other hand, it reduces the time of drawing very materially. The enlarged size of the door makes drafting more difficult and leads to much burned coke. The practice of watering far in advance of drawing and especially where the precaution of putting the lid on trunnel-head is not

followed, leads to the rapid cooling of the oven and reduces the yield by increasing the length of time necessary for subsequent charges to ignite. This increases the time to burn the same size of charge or necessitates the reduction of the charge to burn ovens down on time.

Lately a patented machine for the mechanical leveling of coal

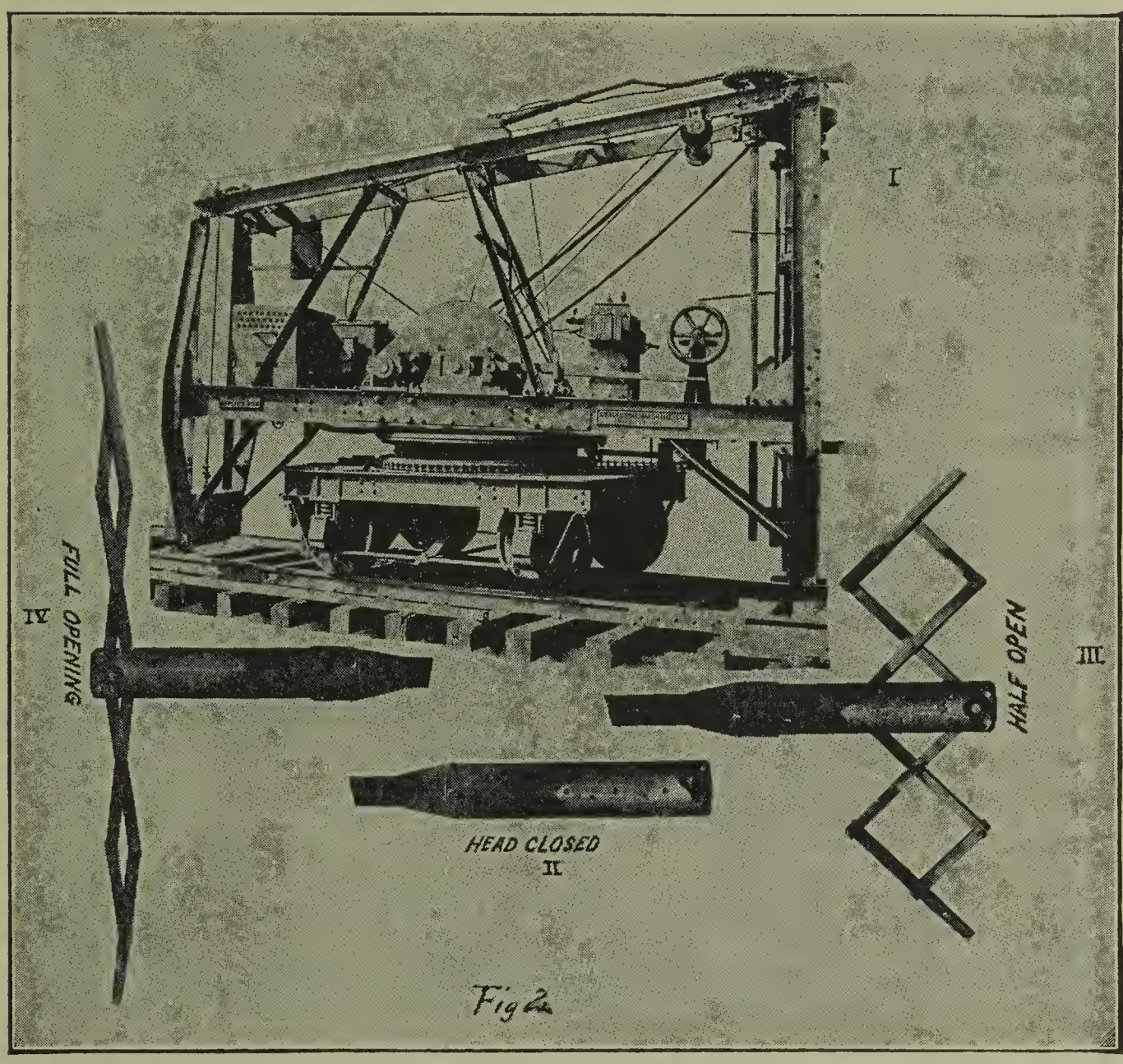


Fig 2.

in beehive ovens has been put on the market and is now in operation in the Connellsville coke region (Figure 2). It runs on the larry track, uses the larry trolley, and operates through the trunnel-head. This machine gives evidence of proving its usefulness, not only by the elimination of hand labor, but by encouraging the proper leveling of the ovens, an operation too often neglected or considered of little importance. The proper leveling of the coal

in any oven is a matter of great importance, and it is a source of regret that so little attention is paid to this feature of the process by the beehive operators throughout the country. With improper leveling, the different heights of charge become coked to the bottom at different periods of time, thus exposing the top surface of the coke to the prolonged action of the air admitted for the completion of the process, with a resultant loss of coke, or if the process be stopped short of completion the coke from the higher portions exhibits black butts.

LONGITUDINAL OVENS

The decreasing efficiency and scarcity of labor and the increased cost of coking coal has led to experiments to reduce both of these factors, first by making the coking operation non-dependent on large numbers of laborers, and secondly by decreasing the cost of actual operation. From these experiments was evolved in 1906 the longitudinal oven (a modification of the old Belgian type), with its mechanical devices for leveling, pushing and loading of coke. Figure 3 shows a section of this type of oven and the method of operation. In its essential details it may be described as a long, narrow, rectangular chamber generally somewhat larger at the discharging end, with sloping barrel roof approaching the center from both ends, a trunnel-head in center of roof, flat tile bottom and doors the whole width of the chamber at either end. These ovens are placed side by side forming a block and are charged as in beehive practice, from a larry running on a track supported on the ovens. The oven is drafted from both ends, after the method of the beehive oven, and the coke, after being watered on the inside of the oven, is pushed out by means of a pusher, devised for the purpose, onto a traveling conveyor, which transfers it to cars; screening being more or less fully accomplished during the passage of the coke along the conveyor. During the last three years this type of oven has come into prominence, especially in the Lower Connellsville region, and many claims are made for it in regard to increased yield, better product, lower cost of production, etc., but judgment must be withheld until these ovens have been in use for a longer period and prove their worth by actual service.

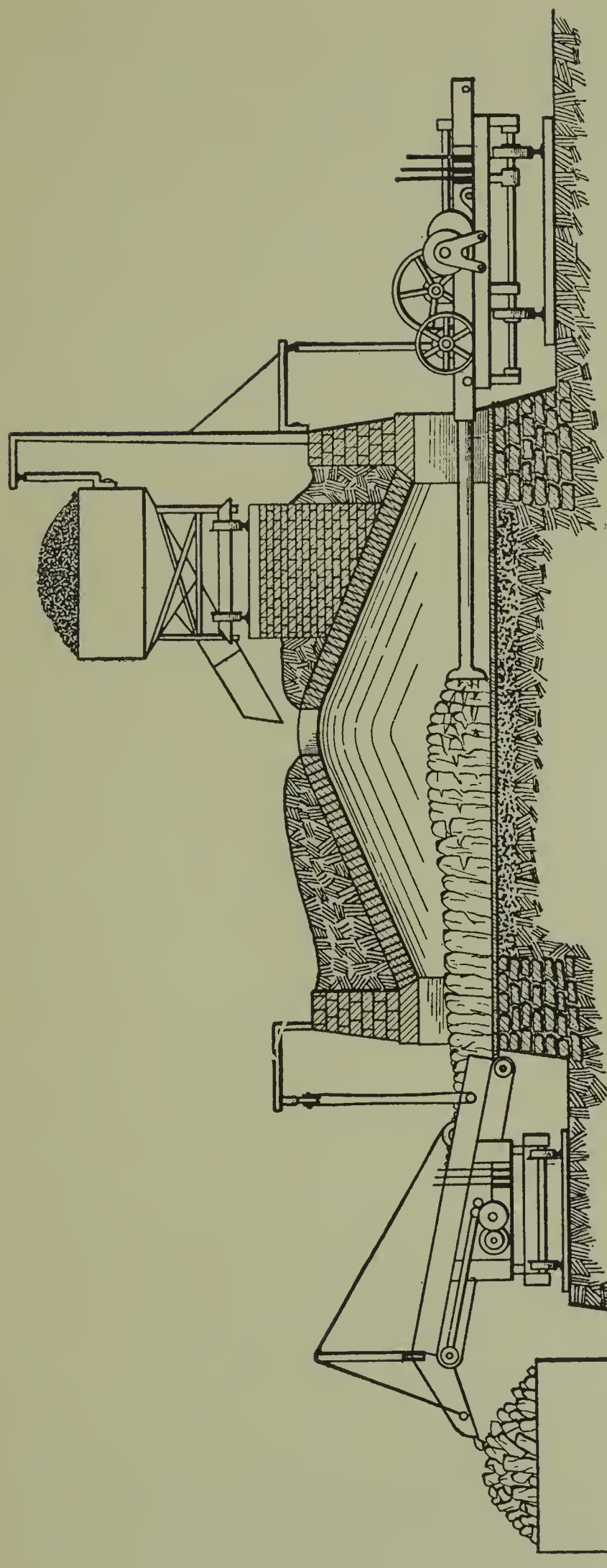


FIG. 3

LONGITUDINAL OVEN

SECTION SHOWING METHOD OF OPERATION

BEEHIVE OVEN WITH ARRANGEMENT FOR UTILIZING WASTE HEAT OF GASES

Waste heat from beehive ovens is utilized at some few plants in this country for the generation of steam. The ovens, either bank or double-row, are provided with a tunnel, built in case of the bank ovens directly behind, and the double-row between the rows, and extending the whole length of the ovens to be used for this purpose. From each oven a flue is provided leading from the oven to the tunnel and so arranged that it can be dampered off. This flue is placed in the crown of the oven above the height to which the coal is charged. Numerous schemes for placing the flues leading from the oven to the main flue have been tried. The usual practice is a short straight flue from the oven direct to the tunnel. At the boiler end a large stack is placed to cause proper draft. In operation the regular trunnel-head is covered and kept closed, the gases passing through the flue into the tunnel instead of into the open air as in the case of the ordinary beehive practice. The waste heat passes along the tunnel in the direction of the draft chimney and through the boiler setting before passing out of the stack. The temperature in these flues is very high (around 2700° F.) and the building of flues to withstand this heat is a matter of much consequence. The temperature of the gases at the boiler approximates 2000 ° F., and the gases are sufficient in amount to generate from 12 to 20 horse power per oven. The ovens burn hotter under these conditions due to the better draft on the ovens than when burning into the air.

BEEHIVE COKING IN THE SEVERAL COAL FIELDS OF THE UNITED STATES

Beehive coke is produced in five of the seven great coal fields of the United States: Appalachian field in Pennsylvania, Virginia, West Virginia, Ohio, Tennessee, Georgia, Alabama and Eastern Kentucky; the Eastern Interior field in Illinois, Indiana and Western Kentucky; the Western Interior field in Kansas, Missouri and Oklahoma; the Rocky-Mountain field in Colorado, Montana, Utah and New Mexico; and the Pacific Coast field in Washington.

Pennsylvania has from the beginning of coke making in the

United States maintained the supremacy as to production, and the beehive oven coke from the Connellsville region is still considered the standard coke of the country. All other cokes from whatever section of the country are judged by comparison with this standard. The coal from this region, with approximately thirty-one per cent of volatile matter, seems to contain just the proper amount and composition of this volatile and to be given off at the proper temperature and time to produce a maximum yield of coke with a minimum loss of fixed carbon during the process. Practically all the coal in this region is mined for the production of coke and is charged into the ovens without any preparation whatever. About fourteen per cent of the coal from other coking regions of the State is washed before coking, and the coke produced is of varying purity and physical structure. The table herewith shows the range of composition of the Pennsylvania cokes:

Moisture	0.23	to	0.91
Volatile matter	0.29	to	2.26
Fixed Carbon	92.53	to	80.84
Ash	6.95	to	15.99
Sulphur	0.81	to	1.87

West Virginia coal is of exceptional purity and the coking industry, although second in production, is mainly as an incident to the furnishing of steam and domestic coal. More than sixty per cent of the coal charged into the beehive ovens is slack and less than ten per cent of it is washed. At those plants where proper attention is given to preparation of the coal and subsequent operation of the ovens themselves, a coke of superior chemical and physical properties is produced. The quality of the greater amount of West Virginia cokes is exceptionally good, but a wide variation is still to be found in the chemical composition of the cokes of this State as the following range shows:

Moisture	0.07	to	0.60
Volatile matter	0.46	to	2.35
Fixed carbon	95.47	to	84.09
Ash	4.00	to	12.96
Sulphur	0.53	to	2.26

The coke produced in Virginia is from coal in the southwestern part of the State and is all from unwashed coal. In chemical and

physical properties it resembles the cokes from southern West Virginia.

The cokes from the southern part of the Appalachian field, comprising the States of Tennessee, Georgia, Alabama and Eastern Kentucky, are for the most part made from washed coal, the quality of coke is poorer and the ash and sulphur are high. The average ash is in the neighborhood of 14 or 15 per cent, and often running up to 16 and 18 per cent where proper attention is not paid to preparation and coking. The Birmingham district of Alabama produces a fairly large amount of good beehive coke, with an ash content averaging 11 per cent, but such coke is the exception and not the rule.

Range of composition of above coke:

Moisture	0.75	to	1.34
Volatile matter	0.75	to	1.95
Fixed carbon	91.20	to	77.81
Ash	7.30	to	18.90
Sulphur	0.58	to	1.77

Beehive coking operations in the States of Illinois and Indiana are practically at a standstill. A few ovens for experimental purposes have been built in these States and have demonstrated the practicability of producing coke from some of these coals. The coking possibilities of Illinois and Indiana coals is confined to small areas, and beehive operations will hardly be established in these fields.

Kansas, Missouri and Oklahoma have had poor success in the production of coke and the total output from these States is very small.

Practically all the coke from the States of Colorado and New Mexico is made from coal previously washed and crushed before charging into the ovens. A plant of 350 beehive ovens of concrete construction is one of the novelties of this region. The ovens proper are of the ordinary beehive type, 13 feet in diameter and 7½ feet in height, constructed of firebrick and tile. The ring walls and wharf walls are of plain concrete, the battery walls and larry track columns of reinforced concrete. The yield of coke is reported above the average for the region and is believed to be due in great measure to the concrete construction which prevents the

entrance of excess air so common in cracks developed in ordinary beehive construction. For details of this plant the reader is referred to Mines and Minerals, February, 1910, pages 429 to 432. At another plant in this region the ovens are provided with under-flues which convey the gases from the coal under the oven bottom to a large flue, back of the ovens, which carries it to the power-house. This power plant furnishes all the power for operation of the mines, for ventilation, electric haulage, coal washing and crushing, and steam heat for the company buildings and electric lighting for the entire community. The flues under the oven bottoms serve to hasten the coking and prevent black ends. The operation requires careful watching to prevent the coal from coking up from the bottom as well as down from the top. When the bottoms become too hot and this coking upwards does take place a distinct line of demarcation, where the two operations meet, is seen over the whole charge of coke.

The cokes of these States are uniformly low in sulphur, scarcely ever exceeding .75 per cent, but the ash is high, averaging 16 per cent.

The coals used for manufacture of coke in Utah are practically identical in character with those of western Colorado, but exhibit the surprising characteristic of losing their coking properties and becoming non-coking if kept in storage for any length of time.

Montana produces a very small amount of coke, all of which is used in copper smelters. Attention is called to the fact that the only restriction placed on the coke is that the ash content shall not exceed 20 per cent. This restriction is only complied with when the coal is washed. An average analysis of this coke shows 18.00 to 21.00 per cent ash and over 2.00 per cent sulphur.

Washington is the only State west of the Rocky Mountains that contains coking coal and the area within the State is small. All the coke is made from washed and crushed coal and is of fairly good quality. An average analysis of this coke follows:

Moisture	0.92
Volatile matter	1.50
Fixed carbon	79.58
Ash	18.00
Sulphur	0.52

ACTION OF DISINFECTANTS ON SUGAR SOLUTIONS

By GEORGE P. MEADE, Gramercy, La.

Read at Joint Meeting with the Eighth International Congress of Applied Chemistry, New York City, September 4-13, 1912.

This work was started with the idea of determining the efficiency of chloride of lime as a preservative for sugar solutions. Later the experiments were extended to include the action of formaline, ammonium, fluoride, and a commercial preservative of the cresol variety. The work was done at odd intervals during the last six months under conditions which made an extended investigation impossible. Therefore, the results are somewhat fragmentary.

All polarizations were with alcohol—50% of 95% alcohol—and a minimum of lead subacetate solution.

The addition of the disinfectant was effected by adding 1 c. c. of a solution of proper concentration to 100 c. c. of sugar solution. For instance "chloride of lime 1 : 10,000" means that 1 c. c. of a 1% solution of chloride of lime was added to 100 c. c. of the sugar solution. In the "control without disinfectant" 1 c. c. of sterile water was always added to compensate for the dilution due to the addition of the disinfectant in the treated samples.

Acidities are quoted in number of c. c. of N/10 KOH necessary to neutralize 10 c. c. of solution, phenolphthalein as indicator.

Sterilized flasks stoppered with cotton were used in all the work.

Experiments I and II. Cane syrup from the Triple Effect 28° Bé, was infected with a portion of a sucrose peptone culture of bacillus vulgatus. The artificial infection was probably superfluous. The syrup was divided into 100 c. c. portions and treated

as shown. The formaline, 1 : 1000, was employed as a comparison since this is the commonest preservative and the ordinary dilution used in sugar work.

ACTION OF CHLORIDE OF LIME ON ARTIFICIALLY INFECTED CANE SYRUPS AFTER SIX DAYS' INCUBATION AT 35–37° C.

	I			II		
	Pol.	Reducing Sugars.	Acidity.	Pol.	Reducing Sugars.	Acidity.
Original	.....	.....	.....	38.7	5.03	
Control, no disinfectant	39.2	6.15	3.0	38.3	3.13	10.5
Chloride of lime, 1 : 100	.....	.....	.....	37.0	7.32	Sl.alk.
1 : 1,000	21.8	17.52	2.1	37.4	6.03	14.4
1 : 10,000	37.9	4.44	3.2	36.8	5.73	13.5
1 : 100,000	30.1	10.16	3.1	33.7	4.92	13.0
Formalin, 1 : 1000	40.1	5.64	0.7	37.6	5.31	2.0

The chloride of lime used in this and subsequent experiments contained 19.75% available chlorine.

The noteworthy point in both these tabulations is that the treated samples in the majority of cases show greater deterioration than the untreated control. Platings and a microscopic examination of the samples in Experiment II showed no growth with Formaline 1 : 1000, so the deterioration in that sample must have been due to the acidity. Chloride of lime 1 : 100 and 1 : 1000 showed numerous organisms. No difference could be detected between the control and chloride of lime 1 : 100,000 so far as the bacterial examination went. It may be that the artificial infection was a disturbing factor in these two experiments.

EXPERIMENTS III AND IV. In Experiment III juice from sound cane was treated and in IV juice from frozen cane. The incubation period was 65 hours in both cases. There was no artificial infection in these or any of the subsequent experiments on solutions taken from the factory.

EXPERIMENT V. Two sets of samples of the same juice were treated identically. One set was polarized at the end of two days, care being taken to prevent contamination, and again at the end of three days. The second set was polarized at the end of five

ACTION OF CHLORIDE OF LIME ON CANE JUICE 65 HOURS IN INCUBATOR AT 35-37° C.

	III			IV		
	Pol.	Reducing Sugars.	Acidity.	Pol.	Reducing Sugars.	Acidity.
Original	10.7	.....	.....	8.75	.....	3.0
Control, no disinfectant	8.8	1.14	11.4	4.4	2.88	12.0
Chloride of lime, 1 : 200	.....	.....	.....	8.3	2.83	0.2
1 : 1,000	8.4	2.35	1.5	6.8	3.79	2.9
1 : 10,000	4.5	1.94	13.5	2.4	3.26	9.5
1 : 20,000	.....	.....	.....	2.3	3.36	10.0
1 : 100,000	9.5	0.93	9.5	4.75	2.35	10.8
Formaline, 1 : 1000	9.15	3.26	1.9	6.75	4.06	3.2

days. In this experiment the incubation was at room temperature.

ACTION OF CHLORIDE OF LIME ON CANE JUICE AT ROOM TEMPERATURE (23–30° C.)

Polarizations only. Original juice = 9.75 Polarization.
3.2 Acidity.

	After 2 Days.	After 3 Days.	After 5 Days.	Acidity After 5 Days.
Control no disinfectant	8.2	7.4	5.85	12.0 c.c.
Chloride of lime, 1 : 200	9.45	9.30	8.85	0.8
1 : 1,000	8.90	8.55	−1.25	6.0
1 : 10,000	6.30	1.5	−1.75	7.5
1 : 100,000	6.90	3.8	4.2	5.5
Formaline, 1 : 1,000	8.25	8.1	7.05	3.5

The three sets of results obtained on cane juice (III, IV and V) are consistent in that they all show a markedly greater deterioration in the presence of chloride of lime, 1 : 10,000 than in the samples where no disinfectant is used. The acidities and invert sugar determinations give no basis from which definite conclusions can be drawn as to the manner in which the organisms have acted on the sugar. Microscopic examinations and platings on sucrose agar of the juice in Experiment V after two days' incubation show both yeast and bacteria in profusion in the control, and in chloride of lime 1 : 10,000 and 1 : 100,000. There were only a few bacteria, all of the gum forming type, in formaline 1 : 1000, chloride of lime 1 : 200 and 1 : 1000.

The next five experiments dealt with the action of various disinfectants. Chloride of lime, formaline, (containing 40% formaldehyde), ammonium fluoride and a cresol compound designated as "Commercial Disinfectant" were the materials employed.

Cane juice was no longer available as a medium, so the raw sugar washings of the refinery were diluted for the purpose. These washings are the heavy syrups purged from the raw sugar in the first steps of the process. Necessarily they are heavily infected with all the organisms which are on the outside of the raw sugar crystals.

EXPERIMENT VI. Action of various disinfectants on raw sugar washings diluted to 15° Bé., 90 hours' incubation at 33° C.

Original — Pol. 21.8 — Acidity 1.8 c. c.
Control, no Disinfectant — 3.5 — 19.5 c. c.

	Chloride of Lime.		Formaline.		Commercial Disinfectant.	
	Pol.	Acidity.	Pol.	Acidity.	Pol.	Acidity.
1 : 200	8.5	9.0				
1 : 1,000	7.5	17.0	19.2	6.0	21.9	2.0
1 : 10,000	1.8	21.5	0.0	17.0	0.2	17.0
1 : 100,000	1.6	21.0	2.2	20.0	2.4	20.5

EXPERIMENT VII. Action of various disinfectants on raw sugar washings to 15° Bé., 66 hours' incubation at 30°–33°. Polarizations only.

Original Solution 22.2
Control, no Disinfectant 5.4

	Ammonium Fluoride.	Chloride of Lime.	Formaline.	Commercial Disinfectant.
1 : 200	22.2			
1 : 1,000	0.4	2.4	21.2	22.0
1 : 10,000	1.8	5.4	3.0	4.5
1 : 100,000	2.7	.....	3.4	2.9

The chloride of lime used in Experiments VI and VII was found to contain only 7.35% available chlorine.

EXPERIMENT VIII. Action of various disinfectants on raw sugar washings, at 20° Bé., after 2 and 3 days' incubation respectively. Polarizations only.

Original Solution.....27.0
Control, 2 days.....10.7. 3 days, 5.3.

	Ammonium Fluoride.		Chloride of Lime.		Formaline.		Commercial Disinfectant.	
	2 Days.	3 Days.	2 Days.	3 Days.	2 Days.	3 Days.	2 Days.	3 Days.
1 : 1,000	9.5	2.8	12.2	6.1	25.6	25.1	25.4	24.8
1 : 10,000	9.2	4.1	10.2	4.6	12.2	6.7	14.5	10.3
1 : 100,000	10.5	5.1	12.1	6.1	8.4	3.2	9.3	4.3

EXPERIMENT IX. Action of formaline on raw sugar washings, 20° Bé. Four days' incubation at 33°-35° C.

Sample.		Polarization.
Original		30.9
Control, no Disinfectant		15.8
Formaline	1 : 1,000	24.1
	1 : 5,000	16.7
	1 : 10,000	15.8
	1 : 20,000	14.6
	1 : 50,000	11.2
	1 : 100,000	16.2

EXPERIMENT X. Action of various disinfectants on raw sugar washings, 15° Bé., 60 hours' incubation at 30°–35° C.

Original 24.1
Control 13.0

	Chloride of Lime.	Ammonium Fluoride.	Formaline.
1 : 1,000	11.8	14.7	19.6
1 : 5,000	11.7	14.8	18.2
1 : 10,000	11.7	14.4	16.7
1 : 20,000	12.3	.....	10.6
1 : 50,000	.....	.....	12.1
1 : 100,000	.....	.....	12.2

Microscopic examination of some of the samples in Experiment X showed yeasts and bacteria to be about equally numerous in the control, in formaline 1 :5,000 and 1 :50,000 and in Chloride of lime, 1 : 10,000. Ammonium fluoride 1 : 1,000 showed yeasts in abundance but few bacteria. All of the samples including formaline 1 : 1,000 contained much gas.

The foregoing experiments all show that the disinfectants dealt with actually aid deterioration when present in sugar solutions in small amounts.

Ammonium fluoride is employed in distilleries in the proportion of 4–8 grams per hectolitre (about 1 : 10,000) for the purpose of inhibiting the growth of butyric acid bacteria without preventing the development of yeast. The fluoride stimulates the decomposing power of the yeasts when present in certain proportions. ("Sugar and the Sugar Cane," Noel Deerr, page 367.)

Since the results with other disinfectants were of the same general character as those obtained with ammonium flouride, it seemed possible that the action was similar, although microscopic examinations had failed to indicate that such was the case.

Experiments with Disinfectants in Artificially Infected Sucrose Peptone Solutions

The medium employed for these experiments was the same as that described by Owen, "Bacterial Deterioration of Sugars" (Louisiana Bulletin No. 125).

Peptone	0.10%
Sodium Phosphate	0.20
Potassium Chloride	0.50

The percentage of sucrose was varied in the different experiments.

Experiment XI. One portion of a sucrose peptone solution containing 10% sucrose was inoculated with a pure culture of yeast; a second portion with bacillus vulgatus. These solutions were incubated for 24 hours. They were then transferred in 100 c.c. portions to sterilized flasks, care being exercised to avoid contamination. Chloride of lime was then added in various pro-

portions. The "original polarization" is on the infected solutions after the twenty-four hours' incubation.

ACTION OF CHLORIDE OF LIME ON INFECTED SUCROSE, PEPTONE SOLUTION (10% SUCROSE) 3 DAYS' INCUBATION

	Yeast.	Bacillus Vulgatus.
Original	5.5	7.6
Control, no disinfectant	0.5	2.9
Chloride of lime, 1 : 200	5.2	7.4
1 : 1,000	Unable to clarify	Unable to clarify
1 : 10,000	0.9	Unable to clarify
1 : 100,000	−1.0	−1.1

EXPERIMENT XII. Action of chloride of lime on infected sucrose peptone solution (20% sucrose) 3 days' incubation. Room temperature.

	Yeast.		Bacillus Vulgatus.		Mixture of Yeast and Bac. Vulgatus.	
	Pol.	Acidity.	Pol.	Acidity.	Pol.	Acidity.
Original	16.1	1.6	17.0	0.45	16.4	1.7
Control, no disinfectant	12.1	1.3	3.1	0.6	12.2	1.3
Chloride of lime, 1 : 200	15.7	Neut.	16.5	Neut.	16.5	Sl. alk.
1 : 1,000	15.0	0.4	16.2	0.2	15.4	Sl. alk.
1 : 10,000	11.9	1.40	2.6	0.5	12.3	1.3
1 : 100,000	12.1	1.0	3.0	0.6	11.6	1.45

EXPERIMENT XIII. Another experiment using chloride of lime with yeasts and bacteria was carried out in 30% sucrose peptone. For the bacteria in this experiment a pure culture of a gum-forming organism isolated from a Cuban raw sugar was used. The organism corresponded in all particulars to the "Bacillus D" described in Lewton-Brian and Deers' Bulletin on the "Bacterial Flora of Hawaiian Sugars."

ACTION OF CHLORIDE OF LIME ON INFECTED SUCROSE PEPTONE SOLUTION (30% SUCROSE). THREE DAYS' INCUBATION. ROOM TEMPERATURE

	Yeast.	Bacillus "D."
Original	30.0	30.0
Control, no disinfectant T.	27.7	21.0
Chloride of lime, 1 : 1,000	26.7	23.7
1 : 5,000	29.8	16.6
1 : 10,000	30.0	21.4
1 : 20,000	29.6	24.9

The experiments with yeasts and bacteria in pure culture with chloride of lime gave results fairly consistent with those obtained on solutions from the factory. There is nothing in these experiments to indicate that the action of the chloride of lime is the same as that of the ammonium fluoride. In fact the solutions containing bacteria gave more positive results, so far as stimulating action is concerned, than those with yeasts. The results are not very conclusive however as the stimulating action of the chloride of lime is not so marked (except in Experiment XIII) as it was where factory solutions were employed.

EXPERIMENT XIV. Experiments with yeast and bacteria were run using formaline as the disinfectant.

ACTION OF FORMALINE ON INFECTED SUCROSE PEPTONE SOLUTION (20% SUCROSE) AFTER TWO DAYS' AND THREE DAYS' INCUBATON RESPECTIVELY

	Yeast.		Bac. Vulgatus.	
Original	16.7		16.7	
	2 days	3 days	2 days	3 days
Control, no disinfectant	12.5	9.8	4.5	3.4
1 : 5,000	16.7	15.5	16.7	16.6
1 : 10,000	14.4	11.8	16.6	16.4
1 : 20,000	13.8	11.1	16.7	15.3
1 : 50,000	13.4	10.6	5.1	3.4

EXPERIMENT XV. Action of formaline on sucrose peptone inoculated with a mixture of yeasts and bacillus vulgatus. At room temperature.

	Yeasts and Bacteria Mixed.	
Original	15.95	
	2 Days' Incubation.	3 Days' Incubation.
Control, no disinfectant	7.7	4.25
Formaline, 1 : 1,000	12.4	9.70
1 : 5,000	10.3	6.80
1 : 10,000	11.5	8.2
1 : 50,000	9.2	5.4
1 : 100,000	9.5	5.6

The two experiments with formaline in artificially infected sucrose peptone solution failed to show any stimulating action whatever.

In order to see whether the unsatisfactory results so far obtained with infected sucrose peptone solutions were due to the inoculation or to the medium, four experiments were carried out as follows:—

Sucrose peptone solution in 100 c.c. portions was infected with one c.c of a 10% solution of raw sugar washings such as had been used as a medium in previous experiments.

EXPERIMENT XVI. Action of formaline on sucrose peptone solution (30% sucrose) infected with 1% of a 10% raw sugar washing solution, 30°–33°C.

	2 Days' Incubation.	4 Days' Incubation.	Acidity at End of 4 Days.
Control, no disinfectant	19.1	10.2	17.0 c.c.
Formaline, 1 : 5,000	23.5	19.4	5.8
1 : 10,000	23.5	15.8	6.8
1 : 20,000	19.7	12.3	4.0
1 : 50,000	20.0	12.5	17.8
1 : 100,000	19.2	10.8	19.6
1 : 200,000	19.7	11.9	14.7

EXPERIMENT XVII. Action of various disinfectants on sucrose peptone (30% sucrose) infected with 1% of 10% solution of raw sugar washings. Four days' incubation. 30°–33° C.

Original = 28.2.

	Amm. Fluoride.		Formaline.		Chloride of Lime.	
	Pol.	Acidity.	Pol.	Acidity.	Pol.	Acidity.
Control, no disinfectant...	25.3	3.1	25.3	3.1	25.3	3.1
1 : 1,000.....	27.8	1.8	28.6	0.8	28.2	0.2
1 : 5,000.....	.....	.....	25.9	1.9	26.8	4.4
1 : 10,000.....	21.5	3.2	24.9	2.1	22.5	4.4
1 : 20,000.....	.....	.....	24.7	2.5	26.5	5.0
1 : 50,000.....	18.0	3.2	23.8	2.4		
1 : 100,000.....	.....	.....	21.5	2.9		

EXPERIMENT XVIII. Action of various disinfectants on sucrose peptone (30% sucrose) infected with 1% of 10% solution of raw sugar washings. Three days' incubation. 30°–35° C.

Original = 28.3.

	Amm. Fluoride.	Formaline.	Chloride of Lime.
Control, no disinfectant....	27.9	27.9	27.9
1 : 1,000.....	28.3	.....	28.0
1 : 5,000.....	25.0	.....	27.0
1 : 10,000.....	24.8	26.5	27.4
1 : 20,000.....	.....	27.5	28.6

EXPERIMENT XIX. Action of various disinfectants on sucrose peptone (20% sucrose) infected with 1% of 10% solution of raw sugar washings. Three days' incubation at 33°–35° C.

Original = 20.2.

	Amm. Fluoride.	Formaline.	Chloride of Lime.
Control, no disinfectant....	17.5	17.5	17.5
1 : 1,000.....	20.2	20.2	20.0
1 : 5,000.....			
1 : 10,000.....	18.1	20.0	19.4
1 : 20,000.....	13.3	18.9	17.3
1 : 50,000.....	.....	18.1	
1 : 100,000.....	.....	18.7	

Of the four experiments with sucrose peptone inoculated with a small portion of raw sugar washings, two gave results of the same class as those obtained while working with solutions from the factory, while two failed to give such results.

If time had permitted, experiments of the same character as those just recorded, varying the reaction and composition of the medium, the time and temperature of incubation, and various other factors, would have been carried out. This class of work might have given an insight into the conditions under which the stimulation by the disinfectants takes place most readily.

In order to show that there is no chemical action on the part of the disinfectants themselves, sterile sucrose peptone solutions, to which had been added ammonium flouride 1 : 1000 and chloride of lime 1 : 1000 respectively, were kept in the incubator at 38° C. for three days. No change in polarization was observed in either solution.

Summary of Results

I. Chloride of lime, ammonium flouride, formaline and the cresol disinfectant, when present in sugar solutions in small amounts varying with the disinfectant and with undetermined conditions cause a markedly greater deterioration than occurs in untreated samples.

II. Experiments with chloride of lime in pure culture of yeast and gum-forming bacteria indicate that the stimulation occurs in the case of both organisms.

III. Ammonium fluoride one part to one thousand parts of sterile sucrose peptone and chloride of lime in like proportion caused no change in the polarization of the solution during three days' incubation at 38° C.

Note.—Since the foregoing paper was submitted to the Congress there has come to my notice an article by Hugo Kühl in Pharmazeutische Zentrallhalle, Vol. 52, pp. 1316-1317, which has direct bearing on my work. The article is a review of the results of investigations by various men which show that very dilute solutions of poisonous antiseptics increase the growth of bacteria, yeast, moulds and plant life generally. An abstract follows:

Formaldehyde, 1 :500,000 in milk gave a vigorous growth of penicillium glaucum in five days. Without the formaldehyde there was only slight growth in eight days.

Ono found that forty parts per million of copper sulphate doubled the mycelia of aspergillus niger formed in sugar solutions. Zinc sulphate in amounts varying from two parts per million to one hundred sixty parts per million increased growth also.

Shultz showed that mercuric chloride, chromic acid, formic acid and salicylic acid in very small amounts stimulate the growth of yeasts. For example, mercuric chloride 1 : 500,000 gives distinct stimulation. These poisons also stimulate bacterial growth.

In large quantities the action is inhibitory; in very minute quantities there is no action; between these two extremes stimulation occurs. The line of demarcation is not sharp. The amount of stimulation and the dilution at which it occurs is dependent on the presence of other substances. Increase of temperature increases stimulation.

The reason for this stimulating action has not yet been determined.

THE DECOMPOSITION OF LINSEED OIL DURING DRYING

By J. C. OLSEN and A. E. RATNER

Read at the Joint Meeting with the Eighth International Congress of Applied Chemistry, New York, September 4-13, 1912.

There are various statements, in the literature on linseed oil, that during the process of drying carbon dioxide is given off. The authors have failed to find the record of any definite experiment indicating the amount of this constituent which is evolved during the drying process. No definite information could be found with reference to the amount of water evolved. Experiments have been conducted to ascertain the increase in weight of linseed oil during drying, the assumption being that this increase in weight is due to the absorption of oxygen. It is evident that, if volatile constituents are given off during the drying process, the increase in weight will not give a true measure of the oxygen absorbed.

In order to secure more definite information with reference to this very interesting and important reaction, an experiment was carried out in which pure, dry air was conducted over a weighed amount of linseed oil. The increase in weight of the linseed oil was ascertained and the moisture and carbon dioxide given off were absorbed and weighed so that the total amount of oxygen which combined with the linseed oil could be calculated.

The linseed oil used for this purpose was a sample of the oil prepared under the direction of Committee E of the Society of Testing Materials. Four samples were prepared under the direction of this committee under conditions which seem to absolutely guarantee that the samples taken were pure linseed oil. Four samples were received from Mr. G. W. Thompson, sealed and packed exactly as they were sent out by this committee for analysis.

The full description of the method of preparing these samples, as well as the analysis, may be found in the report of Committee

Edward Barrow

D of the Society of Testing Materials. The sample upon which our experiment was conducted was pressed from the seed by the National Lead Company, April, 1909. On the 25th of April, 1912, when our experiment was begun, the oil was clear but there was a slight sediment in the bottle. The bottle was thoroughly shaken when the portion experimented on was weighed out.

5.336 grams of the linseed oil was transerred to a weighed Florence flask of 400 c.c. capacity. In order to expose this large amount of oil in a thin film to the gases of the air 3.8666 grams of glass wool were placed in the bottle. By previous experiment, this amount had been found just sufficient to soak up the oil after the walls of the flask had been covered by a thin film. A similar flask was used as a counter-poise in all the weighings so as to eliminate the error due to air displacement and films of moisture on the surface of the glass.

The flask containing the linseed oil was connected up in a series of tubes as follows. A glass tube extended into the open air so as to avoid acid fumes and impurities from the laboratory air. The air was first passed through a large tower containing soda lime and caustic potash in lumps, then through a Geissler bulb containing strong caustic potash solution; then through a U tube containing concentrated sulphuric acid and glass beads. The air thus freed from carbon dioxide or other acid gases and water passed into the flask containing the linseed oil through a tube extending to the middle of the flask. The exit tube passing out from the upper part of the flask conducted the air into a weighed U tube containing concentrated sulphuric acid and glass beads; thence into weighed Geissler bulbs containing strong caustic potash solution; then through a weighed U tube containing concentrated sulphuric acid and glass beads; then through another sulphuric acid tube to an aspirator, holding 7.5 liters. This aspirator drew 7.5 liters of air through the apparatus at night and 7.5 liters during the day; the flask containing the oil, the sulphuric acid tubes, and the Geissler bulb being weighed morning and evening.

Before the experiment was started, the apparatus was tested over a long period of time by drawing air through in the manner indicated, and weighing the tubes night and morning until it was certain that all sources of error had been eliminated and that the various weighed tubes had become constant. The flask designed

for the linseed oil then received its charge of weighed linseed oil, and the experiment continued for 74 days. The results of the experiment are given in tabular form.

The determinations bracketed are doubtful on account of slight errors such as spattering of the liquid in the tubes, etc. Table II was calculated from Table I by adding together the daily increases in weight, so that the figures given for each day give the total quantity on that day. Table III was calculated from Table II by dividing the various quantities by the weight of oil taken, 5.366 grams.

It will be noted that moisture and carbon dioxide were given off almost immediately in fairly large quantity. The oil which was slightly yellow in the beginning soon became colorless and gradually acquired a dark yellow color. Volatile matter also began to be evolved from the linseed oil. This was ascertained from the observation that small oily drops collected in the neck of the flask. The weighed sulphuric acid tube also gradually acquired a brown color which ultimately became black. This would, undoubtedly, produce an evolution of sulphur dioxide, which would be absorbed by the caustic potash solution in the Geissler bulb. Only toward the end of the experiment did the sulphuric acid tube which followed the Geissler bulb acquire a dark color. It is reasonable to suppose, therefore, that little, if any, volatile matter escaped absorption and that, therefore, while the increase in weight of the absorption tubes does not correctly represent the amount of water and carbon dioxide evolved, it does represent the total volatile matter given off by the oil, so that the increase in weight of the oil, plus the increase in weight of the absorption tubes represents the total amount of oxygen absorbed by the oil, and that experiments in which only the increase in weight of the linseed oil film is noted, do not represent correctly the amount of oxygen absorbed. On the accompanying plate, a curve is drawn representing the amount of oxygen absorbed in the reaction. This was obtained from the increase in weight of the oil, plus the increase in weight of the absorption tubes. Another curve is given, showing the amount of water evolved, and another one showing the amount of carbon dioxide evolved. The accuracy of these curves and more especially the one representing the amount of water evolved, is vitiated by the fact that a volatile organic substance was produced. This volatile substance, however,

TABLE I

Daily Record of Weights Taken

Days.	Increase in Weight of Oil, Grams.	Water Given Off, Grams.	Carbon Dioxide Given Off, Grams.
1st	0.0254	0.0306	0.0156
2d	0.0060	0.0156	0.0106
3d	0.0044	0.0236	0.0138
4th	0.0060	0.0316	0.0244
5th	0.0102	0.0088	0.0270
6th	0.0140	0.0118	0.0124
7th	0.0178	0.0334	0.0080
8th	0.0286	0.0164	0.0038
9th	0.0276	0.0218	(0.0084)?
10th	0.0156	0.0102	0.0128
11th	0.0374	0.0292	0.0094
12th	0.0454	0.0364	0.0072
13th	0.0274	0.0202	0.0063
14th	0.0696	0.0438	0.0095
15th	0.0796	0.0246	(0.0074)?
16th	0.0756	0.0200	0.0054
17th	0.0650	0.0098	0.0000
18th	0.0478	0.0156	0.0078
19th	0.0600	0.0230	0.0066
20th	0.0636	0.0150	0.0042
21st	0.0408	0.0270	0.0058
22d	0.0354	0.0212	0.0056
23d	0.0400	0.0560	(0.0056)?
24th	0.0184	0.0272	0.0057
25th	0.0112	0.0356	0.0079
27th	0.0114	0.0268	0.0052
28th	0.0108	0.0088	0.0010
30th	0.0156	0.0260	0.0090
32d	0.0064	0.0304	0.0022
35th	0.0082	0.0202	0.0078
38th	0.0058	0.0130	0.0028
41st	0.0092	0.0046	0.0028
44th	0.0071	0.0061	0.0035
48th	0.0054	0.0057	0.0038
51st	0.0058	0.0072	0.0018
53d	0.0040	0.0086	0.0026
57th	0.0032	0.0043	0.0020
62d	0.0012	0.0068	0.0025
74th	0.0011	0.0052	0.0007

TABLE II

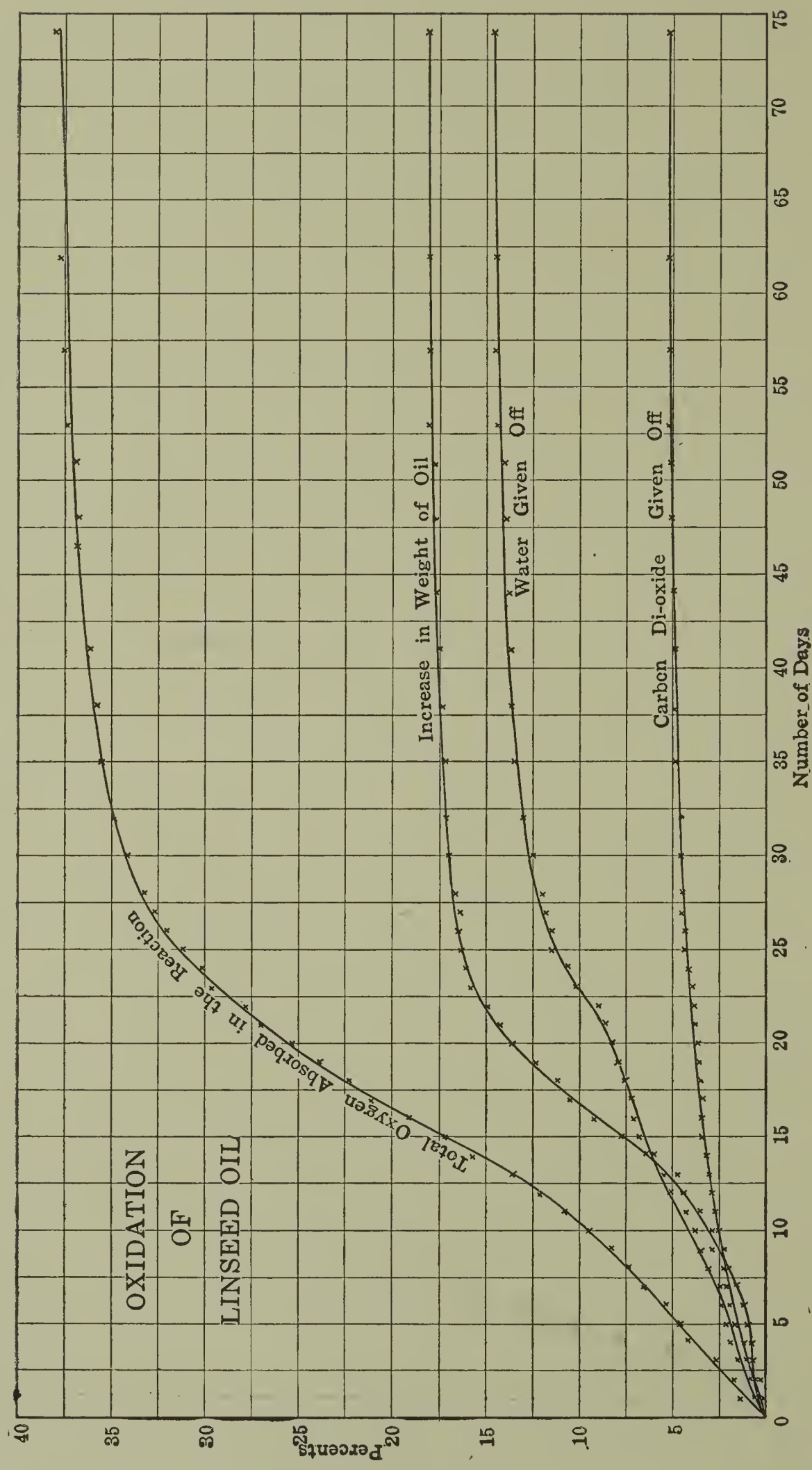
Total Amount of Quantities Determined

Days.	Increase in Weight of Oil Grams.	Water Given Off, Grams.	Carbon Dioxide Given Off, Grams.	Total Oxygen Absorbed, Grams.
1st	0.0254	0.0306	0.0156	0.0716
2d	0.0314	0.0462	0.0262	0.1038
3d	0.0358	0.0698	0.0400	0.1456
4th	0.0418	0.1014	0.0644	0.2076
5th	0.0520	0.1102	0.0914	0.2536
6th	0.0660	0.1220	0.1038	0.2918
7th	0.0838	0.1554	0.1118	0.3510
8th	0.1124	0.1718	0.1156	0.3998
9th	0.1400	0.1936	0.1240	0.4576
10th	0.1556	0.2038	0.1368	0.4962
11th	0.1930	0.2330	0.1462	0.5722
12th	0.2384	0.2694	0.1534	0.6612
13th	0.2658	0.2896	0.1597	0.7151
14th	0.3354	0.3334	0.1692	0.8381
15th	0.4150	0.3580	0.1766	0.9496
16th	0.4906	0.3780	0.1820	1.0506
17th	0.5556	0.3878	0.1820	1.1254
18th	0.6034	0.4034	0.1890	1.1958
19th	0.6634	0.4264	0.1964	1.2862
20th	0.7270	0.4414	0.2006	1.3690
21st	0.7678	0.4684	0.2064	1.4426
22d	0.8032	0.4896	0.2120	1.5048
23d	0.8432	0.5456	0.2176	1.6064
24th	0.8616	0.5728	0.2233	1.6577
25th	0.8728	0.6084	0.2312	1.7124
27th	0.8842	0.6352	0.2364	1.7558
28th	0.8950	0.6440	0.2374	1.7764
30th	0.9106	0.6700	0.2464	1.8270
32d	0.9170	0.7004	0.2486	1.8660
35th	0.9252	0.7206	0.2564	1.9022
38th	0.9310	0.7336	0.2592	1.9238
41st	0.9402	0.7382	0.2620	1.9404
44th	0.9473	0.7443	0.2655	1.9571
48th	0.9527	0.7500	0.2693	1.9720
51st	0.9585	0.7572	0.2711	1.9868
53d	0.9625	0.7658	0.2737	2.0020
57th	0.9657	0.7701	0.2757	2.0115
62d	0.9669	0.7769	0.2782	2.0220
74th	0.9680	0.7821	0.2789	2.0290

TABLE III

Total Amount of Quantities Determined in Percentage of Oil Taken

Days.	Increase in Weight of Oil, Per Cent.	Water Given Off, Per Cent.	Carbon Dioxide Given Off, Per Cent.	Total Oxygen Absorbed, Per Cent.
1st	0.47	0.57	0.29	1.34
2d	0.59	0.86	0.42	1.94
3d	0.67	1.30	0.74	2.72
4th	0.78	1.88	1.20	3.97
5th	0.97	2.06	1.70	4.72
6th	1.23	2.27	1.98	5.44
7th	1.56	2.90	2.08	6.55
8th	2.09	3.20	2.16	7.45
9th	2.61	3.61	2.31	8.52
10th	2.90	3.80	2.55	9.25
11th	3.60	4.34	2.73	10.65
12th	4.44	5.02	2.86	12.30
13th	4.76	5.40	2.98	13.30
14th	6.25	6.21	3.16	15.60
15th	7.73	6.67	3.29	17.70
16th	9.14	7.05	3.45	19.60
17th	10.35	7.21	3.45	21.00
18th	11.21	7.50	3.52	22.30
19th	12.31	7.84	3.66	24.00
20th	13.51	8.23	3.74	25.50
21st	14.30	8.72	3.85	26.95
22d	14.94	9.20	3.95	28.00
23d	15.70	10.18	4.05	29.50
24th	16.00	10.67	4.16	30.90
25th	16.25	11.30	4.30	31.90
27th	16.41	11.80	4.41	32.70
28th	16.65	12.00	4.43	33.10
30th	16.95	12.50	4.60	34.00
32d	17.10	13.05	4.64	34.80
35th	17.21	13.41	4.78	35.50
38th	17.35	13.66	4.83	35.80
41st	17.50	13.73	4.89	36.20
44th	17.65	13.82	4.95	36.50
48th	17.75	13.95	5.03	36.80
51st	17.80	14.10	5.05	37.00
53d	17.90	14.26	5.10	37.30
57th	17.95	14.35	5.14	37.50
62d	18.00	14.48	5.20	37.70
74th	18.05	14.55	5.21	37.80


OXIDATION
OF
LINSEED OIL
Total Oxygen Absorbed in the Reaction
Increase in Weight of Oil
Water Given Off
Carbon Di-oxide Given Off
Percents
Number of Days
0
5
10
15
20
25
30
35
40
45
50
55
60
65
70
75

is probably high in hydrogen, and an attempt will be made in the future to isolate a larger quantity of this constituent and identify it.

The composition of linseed oil is generally given as follows: Carbon 76 per cent; Hydrogen 11 per cent; Oxygen 13 per cent.

Neglecting the error due to the volatile oil given off, the linseed oil lost 1.87 per cent of its carbon and 14.73 per cent of its hydrogen.

At the time this paper was written, the flask and the absorption tubes had not yet become constant in weight. The experiment will be continued until constant weight is obtained.

It was believed that the results, though incomplete, were of sufficient interest for publication.

TESTS ON THE OPACITY AND HIDING POWER OF PIGMENTS

By G. W. THOMPSON.

Read at the Detroit Meeting, December 4, 1912.

In the discussion of paint problems, certain terms are often used with such different meanings that great confusion has resulted. Thus the phrase "covering power" is defined in three or more different senses by Dr. Dudley in his articles in the Railroad and Engineering Journal running in the issues of 1890 to 1893; and the word "body" has so many different meanings that hardly two persons consider it as referring to the same thing. For this reason it seemed desirable to Committee D 1 of the American Society for Testing Materials that the use of these two terms should be discouraged; and they have substituted two simpler terms to cover the more usual uses of these words. These terms and their definitions are as follows:

Hiding Power: The power of a paint or paint material, as used, to obscure optically a surface painted with it.

Opacity: The obstruction to the direct transmission of visible light afforded by any substance, comparison being made with sections of equal thickness. The opacity in the case of pigments should be considered as referable to their opacity in a vehicle under standard conditions.

The distinction between opacity and hiding power should be evident in the study of these definitions. Opacity refers to tests made under standard conditions; and hiding power refers to tests made of paints, etc., as they are used. The distinction becomes clearer when considered with reference to a paint the opacity of which is measured with a standard thickness of paint, while in the case of the hiding power the thickness of the paint will vary according to the spreading rate at which the paint is applied.

In many laboratories tests for opacity have been conducted

on the assumption that what is known as the strength or tinting strength of a pigment is a measure of its opacity. From numerous tests which we have made we have come to the conclusion that strength is an indication only of opacity, and that working on pigments of the same composition, it is not safe to assume that the strength of the pigment is a measure of its opacity. By *strength* or *tinting strength* we mean here:

The relative power of coloring a given quantity of paint or pigment selected as standard for comparison, which is the definition agreed upon by Committee D 1 of the American Society for Testing Materials. Much heated discussion has appeared in the Farben-Zeitung during the last year or more as to whether strength is proportional to opacity. Unfortunately, these discussions are largely academic and not based on practical or accurate tests. As far as the discussions go, it would appear that they have not led to any definite conclusion.

Without going into the question of the tinting strength of pigments in this article, we propose to discuss a method which we have developed for the measurement of the opacity of pigments and paints which will serve, we hope, to some extent, at least, to clear up one phase of this subject.

In developing a method for the determinations of the opacity of pigments, it has been impressed upon us that opacity should never be measured in terms of weight. This has been brought out by Dr. Dudley and some of the disputants in the Farben-Zeitung, but is not generally recognized as it should be. It seems to us that there can be no question but that in all comparisons of opacity, the relative volume of the pigment should be considered and that a standard of opacity should be based upon a definite volume of the pigment placed in a definite volume of a menstruum. The futility of comparing pigments for opacity by weight is evident where these pigments vary in their specific volumes as most pigments of different compositions do.

In comparing pigments or paints for opacity, we are compelled to recognize that it is somewhat of a physiological problem. We really have no good means of detecting differences in light except in the sensations they produce upon the retina of the eye. Photochemical and photoelectrical methods have not so far proved satisfactory. This being the case, all photometric work has to be

based upon certain standards for comparison. In the case of tests for opacity, however, we have not as great difficulty in this respect as we have in the case of the ordinary photometric measurements. By the use of a single source of light and a suitable photometric bench, the opacity of a substance can be determined with a certain degree of accuracy. Following the method used by Hürter and Driffield, who worked upon photographic plates, it is possible to construct plates varying in opacity and whose opacity can be determined. It is hardly necessary to describe in detail the method to be followed for this work, and we would refer to the original article by Hürter and Driffield in the Journal of the Society of Chemical Industry, Vol. IX, 1890, page 455.

There is, however, one difficulty which affects the determination of opacity and the preparation of standard opacity test pieces. In a one light photometer the light is reflected so as to come from opposite directions, and when properly balanced the light should be equal at zero. By placing the object to be tested in the course of one of these beams of reflected light, the light becomes reduced and the balance of light is found at another point which gives a means of calculating the opacity of the object being tested. Unfortunately, the accuracy of the test depends upon no light being reflected by the object being tested, or that the luminosity or reflecting power of the object being tested shall be determined and applied as a correction to the opacity found.

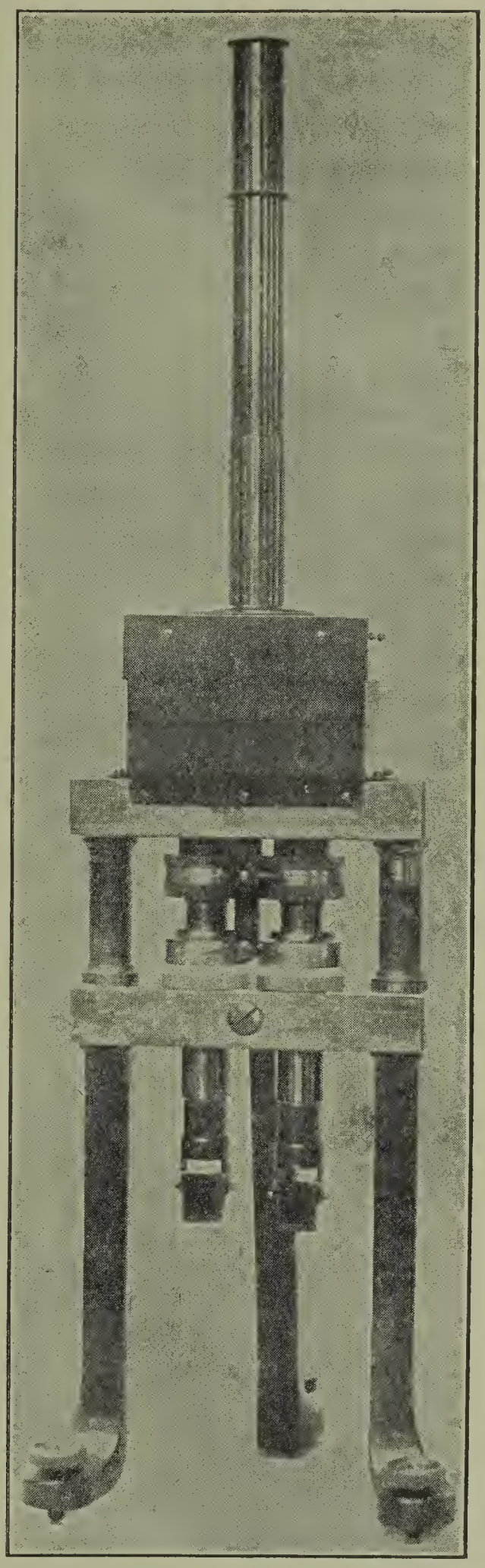
Hürter and Driffield worked upon gelatine silver films which they apparently assumed had no reflecting power, or that, in their case, the reflecting power could be included by them in the opacity for the practical purposes for which tests were conducted. In the testing of white paints, however, this cannot be assumed, for as we will show, the amount of light that is reflected is apparently very much in excess of the light that is absorbed during transmission.

According to the best information obtainable, opacity proper follows a logarithmic law known as Bouguer's Law. Nutting in his recent "Outlines of Applied Optics"—1912, says "Absorption during transmission follows the logarithmic law in every known case; that is, if a given layer absorbs a certain fraction of transmitted radiation, the next equal layer will absorb the same fraction." Thus, if the first layer absorbs half of the light being transmitted, the next layer will absorb half of the remainder of

one-quarter of the light being transmitted; the next layer one-eighth of the light being transmitted and so on.

To express numerically the opacity of a paint, we should define in some term the light absorbed in transmission for a standard thickness. In a paper read before the International Congress of Applied Chemistry, I suggested that this thickness be .1 mm. I find, however, that this thickness is too great for the proper measuring of opacity, and that it would be better to use .01 mm. thickness as the unit of thickness in which to express opacity. Of course, any thickness could be used, but it would be better to have a thickness that corresponds to some practical thickness of paint. I, therefore, feel that it would be better to adopt the latter thickness. In expressing the opacity it would seem to me that it should be called the "coefficient," and having the coefficient we can calculate the total opacity for any given thickness. We would define, therefore, the coefficient of opacity as the proportion of light, expressed in a decimal fraction of unity, absorbed during transmission through a thickness of .01 mm. of paint.

We have constructed a piece of apparatus for the purposes of making these tests which consists, first, of a photometer which will bring two fields of light into juxtaposition



Thompson's Opacimeter.

so that they can be compared by the eye. This photometer is placed on top of two tubes, the lower ends of which are covered with plano-plano lenses. Below these lenses are two other similar lenses. Below these are two total reflection prisms which direct light from a series of incandescent lamps up through the tubes to the eye, and by adjusting the prisms and the lamps, the light can be made to be equally sent up through the two tubes. The tubes holding the upper plano-plano lenses have on them micrometer milled wheels, so that the distance between each set of lenses can be controlled and measured. Paint placed between one set of lenses can be compared with a standard paint or with a piece or pieces of paper which have been tested on a photometer bench and the proportion of light they transmit determined. The thickness of the paint can then be varied until the amount of light transmitted matches that transmitted through the standard paint, or the test papers. The thickness of the film of the paint being tested is then read off on the micrometer. Running another test with a different opacity standard of paper or paint, two readings are obtained from which can be calculated the amount of light that is absorbed and the amount of light that has been reflected. Considering opacity as having to do only with the light that is being transmitted, and not to do with the light which is reflected from the surface of the paint, we can figure the coefficient of opacity by the following calculation:

Let a_1 = proportion of light transmitted by test paper No. 1.
a_2 = " " " No. 2.
$a_1 < a_2$.
b_1 = thickness of paint film transmitting the same amount of light as test paper No. 1.
b_2 = thickness of paint film transmitting the same amonut of light as test paper No. 2.
Same paint formula is used for b_1 and b_2.
$b_1 > b_2$.
c = incident light = unity.
x = proportion of incident light reflected which is independent of the thickness of the film except for very thin films.

Then $\frac{a_1}{a_2}$ = proportion of entering light transmitted by $b_1 - b_2$ thickness of paint, a_2 being the light entering the $b_1 - b_2$ film, as it is the light transmitted by the b_2 film.

It is necessary that we give here the development of a formula for the light that passes through any number of units of thickness of paint:

L = the light passing through any number of thickness units;
S = the light absorbed by any thickness unit or units;
a = the light striking the first surface;

A = the ratio $\frac{L}{a}$;

B = the ratio $\frac{S}{a} = 1 - A$;

n = the number of units of thickness;
P = the constant opacity of each unit of thickness in the form of a decimal fraction of unity.

Light passing through no unit of thickness:

$$L_0 = a \qquad = a.$$

Light passing through one unit of thickness:

$$L_1 = a - Pa \qquad = a(1 - P).$$

Light passing through two units of thickness:

$$L_2 = (a - Pa) - (a - Pa)P = a(1 - P)^2.$$

Light passing through three units of thickness:

$$L_3 = \{(a - Pa) - (a - Pa)P\} - \{(a - Pa - (a - Pa)P\}P = a(1 - P)^3.$$

$$L_n = \qquad a(1 - P)^n.$$

$$\frac{L_n}{a} = A_n.$$

$A_n = (1 - P)^n$. General formula.
$B_n = 1 - A_n = 1 - (1 - P)^n$.

From the formula $A_n = (1 - P)^n$, where A_n is the proportion of entering light transmitted, P is the opacity of unit thickness in terms of decimal of unity, and n is the number of units of thickness.

$$\frac{a_1}{a_2} = (1 - P)^{b_1 - b_2},$$

$$P = 1 - \sqrt[b_1 - b_2]{\frac{a_1}{a_2}}.$$

From formula $B_n = 1 - (1 - P)^n$, where B_n is the proportion of entering light absorbed.

$$B_{b_2} = 1 - \left[1 - \left(1 - \sqrt[b_1 - b_2]{\frac{a_1}{c_9}}\right)\right]^{b_2}.$$

$1-x$=proportion of incident light entering b_2 film,

$$B_{b_2}(1-x)+x+a_2=c=1,$$

$$\left\{1-\left[1-\left(\frac{b_1-b_2\sqrt{a_1}}{1-\sqrt{a_2}}\right)\right]^{b_2}\right\}(1-x)+x+a_2=1,$$

from which

$$x=\frac{1-a_2-\left\{1-\left[1-\left(\frac{b_1-b_2\sqrt{a_1}}{1-\sqrt{a_2}}\right)\right]^{b_2}\right\}}{1-\left\{1-\left[1-\left(\frac{b_1-b_2\sqrt{a_1}}{1-\sqrt{a_2}}\right)\right]^{b_2}\right\}},$$

$$x=1-\frac{a_2}{\left[1-\left(\frac{b_1-b_2\sqrt{a_1}}{1-\sqrt{a_2}}\right)\right]^{b_2}}.$$

This formula seems rather complicated, but in practice and by the use of logarithmic tables the work is more simple than it seems.

The apparatus to which I refer reads to the ten-thousandth of an inch; and preferably should have been constructed with the millimeter scale. It is a simple matter, however, to make conversions into the mm. scale.

In making these calculations it is to be observed that the comparison of the pigments having been made between glass surfaces, the amount of light reflected from the adjacent surfaces of a paint would probably be different from the light reflected from the surface of paint which is adjacent to air. This is a controlling reason why the reflected light should not be considered in calculating the coefficient of opacity.

In testing pigments for their coefficients of opacity, we have followed the plan of mixing these pigmnets with linseed oil on a standard formula of 25 per cent by real volume of pigment and 75 per cent by volume of oil. In some cases, this may be too large a volume for the pigment, as, for instance, in the case of zinc oxide, to handle conveniently in the apparatus; but if trouble is experienced a different formula can be used, comparing it with another standard pigment on this changed formula.

This apparatus is somewhat new and we have not as many results to report of work done upon it as could be desired, and what we present here is simply for information; and so that the

subject will be more generally studied, we present here some determinations made in this apparatus working on a number of white pigments. It is not to be supposed that these tests represent average pigments or that the results presented are for the purpose of condemning any of the pigments tested. It is very probable that the pigments upon the market of the kind described vary considerably from the figures presented herewith.

The coefficients of opacity and the light reflected by the different white pigments tested on the formula given above are shown.

The values for P are the coefficients of opacity as defined above. The reflection is the proportion of incident light reflected and is expressed in decimals of unity.

Pigment.	Coefficient of Opacity, P.	Reflection.
White lead—Dutch	0.0671	0.935
White zinc—American process	0.0794	0.956
White zinc—French process	0.0645	0.964
Lithopone	0.0578	0.947
Calcium carbonate	0.0136	0.969
Basic lead sulphate	0.0813	0.927
China clay	0.0190	0.823
Asbestine	0.0090	0.859
Calcium sulphate	0.0030	0.856
Silica	0.0102	0.793
Barytes	0.0114	0.858

This work was done in the research laboratory of the National Lead Co., and much of it with the assistance of one of my associates, Mr. R. L. Hallett, to whom I tender thanks.

Discussion

President Baekeland: The paper of Mr. Thompson is now open for discussion.

Secretary Olsen: I would like to ask Mr. Thompson, if this $6\frac{7}{10}$ per cent for white lead is $6\frac{7}{10}$ of the total light that goes through?

Mr. Thompson: It is the total light.

Secretary Olsen: That goes through?

Mr. Thompson: That is in process of transmission.

Secretary OLSEN: So that in order to get the per cent of the total light which passes through you had to use your other factor, 93.3?

Mr. THOMPSON: Yes, sir.

Secretary OLSEN: So that it would be about .36 per cent or about a third of a per cent of light goes through.

Mr. THOMPSON: That is, for the coefficient.

Mr. BAKER: I would like to ask Mr. Thompson what proportion of light goes through the linseed oil, if that can be brought under this schedule?

Mr. THOMPSON: We have not made tests on linseed oil; it could not be brought in the test with this apparatus. We have to work with so much thicker films that we would have to construct some different method for determining the opacity or the coefficient of opacity of the linseed oil.

Mr. BAKER: I would like to ask you about the construction of your films.

Mr. THOMPSON: These lenses are detachable, removable from the apparatus. The upper ones are cemented into the upper cylinders, or the expansion of the upper cylinders. The paint is rubbed up as carefully as it can be done without grinding and placed in a little lump, so to speak, on the detachable plate that is shoved into place, and then the micrometer wheel is turned until the two lenses approach contact, then on the other side is a paper that has been found to transmit so much light, so you have a standard proportion of light going through, or you can work with a standard paint, whose opacity or coefficient of opacity you have determined, and then by setting that at a standard thickness you can work the other paint you are testing until the amount of light transmitted in each case is the same as seen through the photometer.

Mr. BAKER: Are these pigments as marketed?

Mr. THOMPSON: Oh, yes, these happen to be pigments that were used in painting a test fence, which has been constructed at Washington, under the auspices of Committee D-1 of the American Society for Testing Materials. The pigments were ground under the direction of the Committee at Pratt Institute. Of course, these particular pigments were ground in the laboratory of the National Lead Co.

Secretary OLSEN: You made no tests of the comparative

opacity of pigments ground fine, of course, so as to get any results on that?

Mr. THOMPSON: No, I didn't want to bring that phase of the subject in. It is a most interesting phase, and we have some work just under way which will give some very, very valuable information regarding the causes of opacity. We naturally think of opacity as being something inherent in the object. Properly we should think of it as due to the presence of non-homogeneous particles. Thus some parts of opaque glass have a higher refractive index than other parts. Apparently there is in the case of very fine particles no such thing as this kind of opacity. Whatever the opacity is it is due to the relation to the medium in which it may be; and we find this, that if the medium has the same refractive index as a particle, then the mixture of the two will be transparent, and that it is due to the difference in the refractive indices that opacity arises. That is one factor in opacity. The next factor that we know of is fineness. The finer the particles, the greater the opacity. Now, whether there are other factors remains to be determined. But, from some other work which we have done, it would appear almost as though those two factors can be considered the dominant factors in opacity with reference to paint and pigments.

Dr. ITTNER: I do not know very much about paints, but the question comes to my mind whether the refractive indices of the different pigments vary much among themselves. Dr. Thompson says that the opacity depends largely upon the difference in the refractive index between the pigments and the vehicle, and the question that comes to my mind is if he had a paint which is made up of two or more pigments, with refractive indices possibly widely divergent from one another, or, as different as possible, whether that would have an influence on the opacity which was appreciable, or whether the difference would be mainly the difference between one of the pigments and the oil itself.

Mr. THOMPSON: There is considerable difference of opinion on one part of the question that Dr. Ittner has asked as to whether in mixed pigments the optical properties are additive, or whether they affect one another. From such work as I have done it would appear that they were additive. Because, you take two paints, one having a coefficient of opacity of six, say, and another paint having

the opacity of two, and you mix them together in equal proportions, you would have a paint that would have an opacity of four. But, there are other elements which come in affecting the hiding power which might make a paint mixture work under the brush very differently and give a greater or a less actual hiding power.

The difficulty which arises in this subject has been a means of determining the opacity of fine particles, and the refractive index of fine particles. I have been trying for years to find some method of determining the refractive index of these particles, but so far have been unable to find such a method. We have some work under way which indicates that we can determine it indirectly. We thought we had a method of determining the refractive index, by discovering that the refractive index bears a very direct relation to the dielectric properties of an object, but as soon as we came to the question of determining the dielectric properties, we found that was harder than determining the refractive index, and we had to give it up. Some give 2.0 as the refractive index of white lead, but where the figure originated I cannot find out, unless by assuming that the refractive index of the mineral cerusite corresponds to the refractive index of white lead. The refractive index of some pigments, such as barytes, are comparatively low, and nearly approach the refractive index of linseed oil, which accounts for their low opacity.

A very interesting thing illustrating this is that calcimine, which is made largely of calcium carbonate, when it is put on with water as the medium, does not cover at all until the water dries out, and air becomes the medium; air having the refractive index of 1.0, calcium carbonate having a refractive index of about 1.5, and water a refractive index of 1.33 the difference becomes very much greater, and a correspondingly increased hiding power is given to the calcium carbonate by the substitution of water by air.

CONTROL OF INITIAL SETTING TIME OF PORTLAND CEMENT

By E. E. WARE.*

Read at the Detroit Meeting, December 4, 1912.

It is well known that Portland cement, as burned in the rotary kiln, is so quick setting that it cannot be used without the addition of some retarding material, such as gypsum or plaster of paris. It is not necessary to make this addition of retarding agent when dealing with the product of a set kiln, probably for the reason that the cement contains the ash of the fuel as well as most of the sulphur.

Occasionally there has been reported a cement of such a nature as to be quick setting even after the addition of the regular amount of retarder, and this paper is in the nature of a report on the commercial manipulation of a 100,000 bbl. lot of such quick setting clinker. The manufacturer was interested, first in correcting the material on hand, and second in establishing a routine of operation that would prevent a recurrence of the trouble.

The setting and hardening of hydraulic mortars has been the subject of considerable investigation, for it is self evident that the quality of the set of a cement determines the ultimate strength of the concrete of which it is a constituent.

Experimenters do not seem to agree very well as to the mechanism of this setting, nor as to the factors that exert the greatest influence during the time that the hydrolysis is taking place. Consequently there is a diversity of opinion as to the method to employ during the processes of manufacture or as to what subsequent treatment the cement must undergo, in order that the manufacturer may at all times put upon the market a cement

*Credit is due to L. C. Nodell and P. H. Chang for the experimental work in connection with this paper.

whose behavior may be predicted, and whose quality will show no deterioration during long time storage.

The consensus of opinion seems to be that the initial set of a cement is due to some action for which the aluminates are responsible, or to which they at least contribute in a large measure.

Also it seems to be quite well agreed that the retarding action of gypsum is due, if not to the formation of a double salt with the aluminates, at least to the fact that it slows down their hydrolysis, and consequently delays the initial set of the cement.

It has been the writer's good fortune to have been, at various times, connected with the operation of Portland cement plants using materials abnormally high in alumina, and he invariably found that it was impossible, under those conditions, to vary the lime content of the cement over any extended range, without precipitating trouble. If the lime was carried high (63-64 per cent), the cement too closely approached the danger line of unsoundness, while if it dropped too low (60.5-61), the factory was troubled with quick setting cement.

Quick setting cement resulting from such operation is not so responsive to the retarding action of gypsum as one more nearly normal in composition. Sometimes it will be quick setting direct from the grinding mills, while at others it will develop a quick set after short storage. Quite often it will show a reversion to quick set if an excess of gypsum is added. The writer had his attention called to a condition where two sections of a plant were operated with differences of 30 per cent in the gypsum added.

None of the cases of quick set in the writer's operating experience ever developed serious difficulties, as the setting times were watched very closely, and at a suspicion of trouble in the stored material, a quick cement was mixed out with a slower one, and preferably one having a tendency toward unsoundness, the combination seeming to remain perfectly stable and not require any further additions of gypsum.

Also at any indication of quick set in the material coming from the mills, the lime in the mix was immediately raised, a procedure that never failed to correct the trouble.

This seems to agree with the experience of Meade,* who states that quick setting cements that have come under his observation

*"Portland Cement," Meade, p. 416.

are low lime cements. He states also, that he has retarded the set of plastered cements that have gone quick by addition of calcium hydrate or even calcium oxide.

It seems to be, however, a direct contradiction to the statements of Reibling and Reyes* who state that all quick setting cements contain free lime, remain quick setting so long as the lime is in the form of oxide, become slow setting as the lime hydrates, again quicken when the hydrate changes at carbonate, and finally become slow setting as the hydraulic constituents become inert through long exposure.

In view of these interesting experiences with quick setting cement, it was with considerable interest that the writer responded to an invitation from a cement company who reported a large stock of clinker as quick setting, and beyond the influence of the ordinary corrective methods.

The clinker was the regular fall run, stored over winter, the plant being one that operated on marl, and followed the usual practice of burning a large stock of clinker during the late fall months to supplement their stock for the early spring demand which opens before the ice leaves the lakes from which they dredge their supply of marl.

An interesting circumstance in connection with the problem is that, although some cement ground in the late fall showed quick setting, the majority of it was perfectly normal except that it would not stand any large additions of quick setting material without itself showing an earlier set. Inquiry developed the fact that this quick setting cement was ground during a short period that the kilns were out of operation, and that when the kilns were started, the rest of the fall grind showed a normal setting time.

Experiments were run to try the effects of different added materials, such as plaster, (instead of gypsum), hydrated lime, calcium chloride, and acids; but none of them seemed to be successful in retarding the set.

At the same time other experiments were tried, along the line of hydration, as recommended by Bamber.† These were highly satisfactory, the cement ground from clinker which showed a set of

*Philippine Journal of Science, 1911, 207.
†Concrete and Const. Eng., 1909 (4) 196.

3-5 minutes under ordinary procedure, being slowed to 2½-3 hours when hydrated to the extent of less than 2 per cent.

It seemed to make little difference how this water was added, as is illustrated by the following experiments:

The cement after grinding with the usual amount of gypsum, was heated in a closed tube, the idea being that the water resulting from the dehydration of the gypsum might prove sufficient for the hydration of the troublesome constituents. This proved to be the case.

The clinker was heated to approximately 100° C. and ground while hot, the result being the same.

The ground cement was dropped through a vertical tube through which a small cloud of steam was rising. Subjection to this atmosphere for even so short a time as that necessary for it to drop through a tube thirty inches long was entirely sufficient to retard the set.

Water to the extent of 3 per cent was added to the ground cement, mixed rapidly by hand, and then placed in laboratory pebble mill where it was mixed mechanically for a short time. The set was delayed, but not so uniformly as by the other methods.

Water was sprinkled on the cold clinker as it was fed to the grinding mills at the factory. This treatment was satisfactory so long as the water supply could be kept constant; but the mill operators could not be depended upon to regulate the supply properly, and the idea was abandoned as impracticable.

Steam was turned into the conveyor leading from the mills. The results from this method were not dependable, and the scheme was dropped as being too uncertain to be safe to use.

The method that was finally adopted was that of heating the clinker and grinding while hot. This method proved entirely satisfactory for the treatment of the greater part of the quick clinker, the remainder being left over until the kilns should be in operation, when the old clinker was ground with the new, the new being purposely not thoroughly cooled.

The adoption of this method was largely influenced by the layout of the plant, which with the clinker pile lying alongside of the kilns and for their full length, made it a simple matter to send part of the clinker through one kiln, and mix it on its return with a quantity of cold clinker. The gypsum used was thoroughly

wetted and added to the clinker just before it reached the mill hoppers, these hoppers being kept only partially filled in order that the clinker might not have time to cool or to dehydrate the gypsum before reaching the mills.

From a consideration of the plant, it was a simple matter to explain the quick setting cement that was ground in the fall. As the stock of clinker grew larger, there was left only one place to discharge the kiln output, and that was at the part of the clinker pile farthest away from the kiln discharge, a point which is nearest to the mills. This meant that for at least the last few weeks the mills were grinding hot clinker; but for the few days that the mills were operated while the kilns were off fire they would be supplied with cold clinker, and so ground out a small amount of quick setting cement.

The table of analyses shows four analyses of quick setting cements, 1-4, and three slow setting cements, 5-7, from the factory stock. The set 8–12 belongs to a series of laboratory cements made from the same raw material, in an endeavor to establish the safe limits for factory operation.

These experimental burns were carried out in a small experimental kiln designed by Prof. E. D. Campbell*, and used by him in all his work on the composition of Portland cement, and the influences that effect its constitution and characteristics.

The clinker from these burns was carefully sorted, and all material that showed any signs of underburning was discarded. The good clinker was then crushed and ground with gypsum.

This set of analyses shows alumina in a fairly high percentage, but fails to show why this alumina has such a decidedly quickening effect under conditions not entirely accounted for by the composition of the cement.

In an endeavor to locate some of the influencing factors, a few further experiments were carried out.

Quenched clinker from these high alumina samples showed quick setting if perfectly dried, but slow setting if only air dried.

Steam clinker when air dried showed a retarded set when ground with plaster.

All cements that have come under observation develop quick set when heated to 350-400° C. This includes a number of com-

*J. Am. Chem. Soc., 24, 248.

TABLE I

Control of Initial Setting Time of Portland Cement

	Clinker.					
	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	No. 6.
Loss	1.0	0.7	2.4	3.0	1.3	0.4
Silica	21.4	22.0	22.4	22.7	22.35	24.9
Ferric oxide	5.1	3.9	3.6	4.0	4.4	4.8
Alumina	6.4	7.0	7.4	6.6	6.35	7.3
Lime	61.0	61.3	61.6	59.8	62.5	61.7
Magnesia	2.3	2.4	2.1	2.3	2.0	2.0
SO_3	1.74	1.57	0.74	1.83	1.57	1.54
$\frac{SiO_2}{R_2O_3}$	1.85	2.02	2.05	2.13	2.07	1.8
Set	quick	quick	quick	quick	normal	normal

	Clinker.					
	No. 7.	No. 8.	No. 9.	No. 10.	No. 11.	No. 12.
Loss	0.03	.....	2.4	.....	.....	1.16
Silica	22.48	24.9	24.4	23.7	23.7	23.9
Ferric oxide	4.2	.....	.....	.....	10.1	9.8
Alumina	7.3	11.7	10.9	10.4		
Lime	62.9	59.4	58.5	61.4	63.3	61.7
Magnesia	2.2	2.11	.....	2.17	.....	2.14
SO_3	1.58	2.21	1.58	1.57	1.36	1.34
$\frac{SiO_2}{R_2O_3}$	1.95	2.13	2.23	2.28	2.36	2.46
Set	normal	quick	quick	quick	3 hrs.	$3\frac{1}{2}$ hrs.

mercial samples of varying composition and compounded from widely different materials. Two commercial cements that had been stored since 1899 and which were presumably in their last stage of slow set, had their initial setting time decreased from 3½ hours to 1 minute. There was a loss in weight during heating of only 0.15 per cent.

A cement with a setting time of 15 minutes was treated alternately with water and heat and showed a setting time curve as in Fig. 1. At each stage in this addition of water and subsequent heating to 350-400° C., a sample of the material was strongly

ignited and showed losses corresponding to the dotted curve of Fig. 1.

From a consideration of the curve it would seem as though the water must have been present in two different conditions, for although the cement showed a continuously increasing amount of water, the set was not correspondingly slowed. This may be partially due to the fact that the heated cement retained some water in such a condition that it was not driven off when heated to 350°

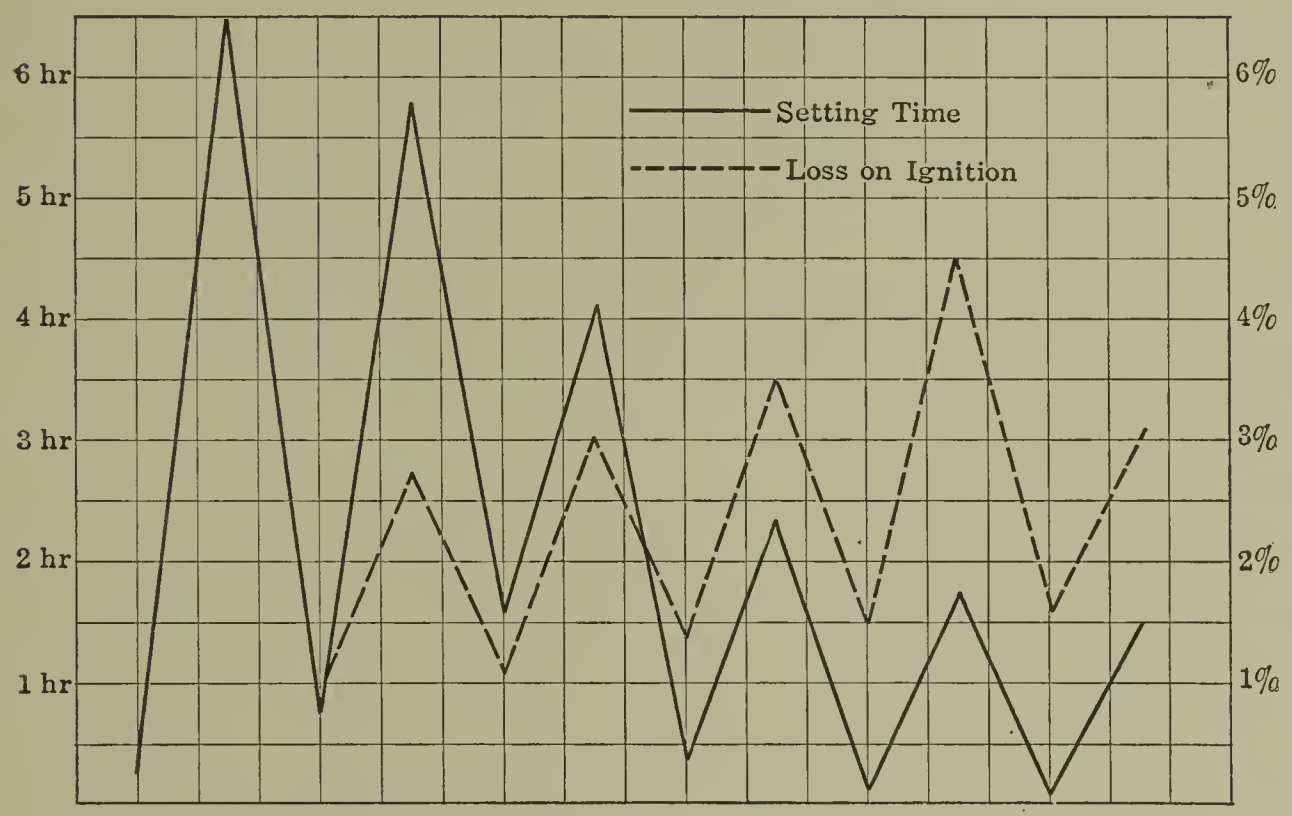


FIG. 1.—Setting Time and Loss on Ignition of Cement.

C., in which condition the water did not seem to exert much influence on the setting time.

In an effort to establish whether it was the gypsum or the cement that was affected by heat, a cement containing no gypsum was heated and afterward mixed with the normal amount. It showed a slow set.

Another sample of the same material, unheated, was mixed with gypsum that had been heated. While it required a larger amount to retard the set (5 per cent)* it showed a normal setting time.

Any one of these slow setting samples would develop a quick set upon heating. In the case of the cement carrying 5 per cent dead burned gypsum, it required a much longer time of heating

*Meade and Gano, Chem. Eng., 1, 292.

than in the case of those samples carrying the smaller amounts. The times of heating varied from 6 hours to 48 hours in the different samples. Dry slaked lime showed no appreciable loss of water under the same conditions of heating.

The whole set of quick setting cements was tested for free lime according to the microscopic method described by Prof. A. H. White.* There seemed to be no indications of free lime.

Although it may be impossible to draw any definite conclusions from this rather incomplete line of experiments, it seems to be certain that in this instance, at least, the quick setting was not due to free lime.

This experimental work is to be continued, in the hope of gathering further data that may assist in clearing away the uncertainties in regard to the role that alumina and gypsum play during the initial setting of Portland cement.

UNIVERSITY OF MICHIGAN,
ANN ARBOR, MICHIGAN.

*J. Ind. and Eng. Chem., 1909 (1), 5.

THE EFFECT OF "LIME SULPHUR" SPRAY MANUFACTURE ON THE EYESIGHT

By JAMES R. WITHROW

Read at the Joint Meeting with the Eighth International Congress of Applied Chemistry, New York, September 4-13, 1912.

About two years ago, the writer was called upon to take charge of the installing of a "Lime-Sulphur" department for a manufacturer engaged in other lines of chemical manufacturing. Preliminary to starting industrial experimentation, a very thorough laboratory study had been carried out for the manufacturer, by his regular chemist. This work reviewed in a most capable manner about all the recommendations, which have recently sprung into chemical and experiment station literature concerning "Lime-Sulphur" preparation. As a result of this work a formula was evolved, which was used as a basis for manufacturing experiments. The laboratory experiments themselves were never made in larger than five-gallon apparatus. The writer witnessed from time to time these experiments or portions of them and at no time noticed anything causing discomfort. The laboratory assistant, who did most of the experimental work for the company's chemist and was constantly in contact with the material and its fumes, never noticed any effect or discomfort at any stage of the laboratory work, which extended through several months. To be sure, there was the ever present odor of hydrogen sulphide or at least a similar odor. This was never offensively strong. At no time was it so noticeable as to compel enforced ventilation.

The writer's business was to accept the work as completed in the laboratory and transfer it to factory operation. The first factory experimental runs were made on about a 12 barrel scale. These experimental cooks were made to get factory scale data for construction work and also to uncover any unforeseen operation

difficulties. The product had a specific gravity, varying from 45° to 32° Bé., depending on the purpose of the experiment. The solution produced of calcium polysulphide or so called "Lime-sulphur," contained about 25 per cent sulphur, and about the equivalent of 10 per cent calcium oxide, when the specific gravity was about 33° Bé. Twelve barrels of this product therefore would contain 1625 lbs. of sulphur and the equivalent of 650 lbs. of lime.

The first few cooks aroused no comment from employees about the building, which was a large one of four stories, beyond what would come from persons unaccustomed to hydrogen sulphide-like odors. In the course of the next week or two, however, the weather had become quite cold and the normal ventilation by means of the windows was much diminished, because of an effort to keep the place warmer by closing the windows. Again no particular effect was noticed at first. The "cook" digester was a steam jacketed cylindrical tank roughly 5′ x 5′ and supplied with a cover and a small ventilating pipe. This pipe was inadequate for proper ventilation of tank and would have been useless anyway, for the top of the "cook" tank was usually always open during the experimental runs. This was for purposes of observation during the experimental cooks. The man in charge of the cooks usually stationed himself at the opening to become familiar with boiling conditions within the tank, during the various runs under different conditions.

Within a cook or two, after the windows were closed to diminish the cold conditions, the man in charge of the cook became aware of a smarting sensation in and around the eyes. The eyelids became red. The writer was constantly about the tank, but was only occasionally at the tank opening and felt little or no discomfort, though there was a slight burning feeling about the eyes. The room became partially filled with condensed steam at times and finally, about 8 P. M., during a run which was a little prolonged, the writer noticed that the steam or vapor in the room was greater than usual and that the incandescent electric lights had a halo of some eighteen inches in diameter, when viewed through the fog. The halo tended to have rainbow colors. An hour or two afterwards, the writer found the same conditions as to fog and halo to exist in his room in his hotel, and concluded that his eyesight

was affected. Cold water was applied liberally and he turned into bed and went to sleep at once. In the morning the blurred eyesight was about as bad as the evening before. The foreman, who stood at the opening of the cook tank, had gone home at the end of the run at the time the writer did. He was unable to report for work next day. His eyes were much inflamed and were too sensitive to light to open them. He said they pained and felt gritty under the eyelids. He was back to work again in a couple of days. In the case of the writer, with the liberal use of saturated boric acid solution the blurred vision gradually returned to normal during the course of a week's absence from the manufacturing operation. There was a recurrence of the blurred effect at another time, which almost rendered vision impossible, but it rapidly wore off and at no time was there any pain. The foreman never again had an attack after his initial experience. None of the workmen were affected after proper precautions were taken.

At one time, however, when a batch was being concentrated by boiling down, the cover was thrown open to expedite evaporation. In the same room some distance away, two workmen were barreling off finished product. Both the foreman and myself were actively engaged about the cook tank and were practically unaffected. Of the two workmen mentioned, however, the thin one was very much affected and said he suffered agony all night and next day, while the corpulent one was entirely unaffected. Other workmen were in and out during the cook, but none were affected. The one of the two mentioned above as unaffected has, since starting regular operation in the new plant and in fact during the rest of the experimental runs, been in active charge of the "cooks" and has never become affected, beyond possibly slight reddening of the eyes.

No one at all has been affected in anyway after the new plant was installed with its ample facilities for ventilation. Inquiry directed to other manufacturers disclosed similar experiences. One manufacturer's experience was so bad that he at once knocked one side out of his cooking room. This is undoubtedly effective, but from the writer's experience unnecessary. All that is required is a hood over the cook tank, which will carry all vapors out doors, and a "cook" room which is high ceilinged and reasonably well ventilated. Providentially the copious evolution of steam has

caused most plants to provide hood-covered tanks, thus avoiding the unexpected trouble we are discussing.

A search of the literature of "lime-sulphur" available to the writer found no mention of the effect on the eyes. The suggested reactions to explain the action of sulphur on calcium hydroxide and water, varied as they were, gave no clue to what might have been the body which gave rise to the trouble. During a subsequent study of polysulphide literature in general, however, it was found that Bloch and his pupils (Ber. d. Chem. Ges., 41, 1961; Am. Chem. Jr., 41, 155) had prepared polysulphides of hydrogen of the formulas H_2S_3 and H_2S_2. The latter is formed by heating the former and is easily volatile. The fumes of these polysulphides are said to have a penetrating disagreeable odor and their vapors attack the mucous membranes. Thorpe says their vapors attack the eyes. (Dict. Applied Chem., 1893, 3, 699). They are decomposed by alkalies and therefore would not exist very long in the lime-sulphur cook, but if they were being given off in mere traces, continuous exposure of such fumes would naturally cause discomfort.

Hydrogen sulphide itself, however, may have been the cause of the trouble. It has been shown to be a product of the evaporation of a solution of calcium polysulphides. (Divers, J. Chem. Soc., 1894, p. 284.) Hydrogen sulphide could not likely have been the cause, except the symptoms of H_2S poisoning recorded are only the effects of sudden or brief exposure to large amounts of the gas and that prolonged exposure to dilute H_2S would cause a different series of violent symptoms. This latter assumption does not appear probable for in such cases where H_2S was permitted in the atmosphere of laboratories in small amounts, the *usual* symptoms, only not so pronounced, were the result. The only recorded symptom of hydrogen sulphide poisoning observed in the cases under discussion was the occasional occurrence of headache. This was to be expected, since hydrogen sulphide was itself being evolved to some extent.

It should be mentioned, however, that K. B. Lehmann (Arch. F. Hygiene, Bd. XIV, 1892, 135; Blyth, "Poisons, Their Effects and Detection," 3d ed., C. Griffin & Co., London, p. 73) mentions cases where "intense irritation of eyes, nose and throat" occurred within five to eight minutes of exposure to a concentration of 0.3

per thousand of hydrogen sulphide, but no affection of the sight is mentioned even in this extreme case. In long exposure to lower concentrations, such as would correspond with the case of hours of exposure in lime-sulphur cooking, the action recorded is on the respiratory tract. These symtoms appeared entirely absent in the lime-sulphur cases as also were all the other common symptoms, (except headache) such as muscular weakness, etc. A tendency to conjunctivitis, a symptom of chronic hydrogen sulphide poisoning, may have been present in the case of the man in charge of the cooks. He was the man, however, whose eyesight itself was never affected. The writer has suffered at other times in the last six years, most of the symptoms of slow hydrogen-sulphide poisoning, due to inadequately ventilated, over-crowded and poorly arranged university laboratories, but the symptoms in the lime-sulphur experience were quite different. In fact the usual muscular weakness and general depression as caused by hydrogen sulphide were not experienced at all in the lime-sulphur manufacture. It should be mentioned also, that the writer has been informed that attendants at "sulphur" baths have had their eyesight temporarily affected in a similar fashion. Volatile polysulphides may be present in this case also, although they have not been proven to be present to the writer's knowledge in either case. This would be an interesting point for someone favorably situated to develop.

It seemed possible therefore that these hydrogen polysulphides might have been the cause of the action on the eyesight of the vapors from the boiling of a mixture of sulphur, lime and water.

It may be stated at this point that this indication of the presence of hydrogen polysulphide in the vapors of the lime-sulphur cooks might have an influence upon the solution of the problem of the actual reactions involved in lime-sulphur preparations, a mooted question at the present time. The trouble with the eyesight always came, when a batch was being concentrated by evaporation before filtration and not during ordinary cooks.

It seemed worth while to record these facts as a warning, at least, as to the serious dangers of lime-sulphur manufacture in the absence of adequate ventilation. This is all the more necessary since it is probable that attention has not already been frequently called to the matter, because ordinary ventilation precautions, only, are necessary to avoid all trouble, and therefore the average manu-

facturer has not had the experience or it has appeared so seldom, that the isolated affection of a workman now and again may have been attributed to something else. It is worth noting also because the mere occurrence of a cold spell of weather gave the opportunity of experiencing this difficulty possible in lime-sulphur manufacture, so that otherwise it might never have occurred at this plant or only in such isolated cases as to lose connection between cause and effect.

LABORATORY OF INDUSTRIAL CHEMISTRY,
OHIO STATE UNIVERSITY,
June 20, 1912.

ACETYLENE SOLVENTS

By J. H. JAMES

Read at the Detroit Meeting, December 6, 1912

PART I.—LABORATORY METHOD AND TESTS

The Laboratory tests made to determine the relative industrial value of various acetylene solvents were carried out as follows:

The acetylene was made from the commercial carbide in an ordinary "Carbide to water" laboratory generator. It was purified to remove ammonia, sulphur compounds, and phosphorus compounds, by passing through a purifying train consisting of the following vessels in order: a 10 per cent sulphuric acid solution, a 15 per cent hydrochloric acid solution saturated with mercuric chloride, two towers containing approximately equal parts of a mixture of bleaching powder and slaked lime, a tower containing slaked lime, only, and finally was completely dried by passing through two towers containing fused calcium chloride.

It is necessary in order to get as closely as possible at the true figure for the absorption of this gas in any of its solvents, that the gas be free from impurities, and that the solvent be of the highest purity attainable. It has been demonstrated that the solubility drops off rapidly when impurities are present, either in gas or solvent. To get the highest commercial efficiency it will pay to purify the gas and select solvents of highest purity. Care with reference to the purity of gas and solvent is at present not given the attention in this industry that it deserves.

The method in detail of carrying out this absorption test was as follows:

A carefully measured volume of the solvent (usually 1.5 c.c.) was placed in an ordinary five-inch side neck test tube, fitted through a two hole rubber stopper with a glass tube gas inlet and a thermometer, the bulb of which was immersed in the solvent.

This tube with solvent was immersed in a freezing mixture (ice and salt) and cooled to —18° C. or —19° C. before starting. The purified gas circulated through a four foot coil immersed in the freezing mixture, thus bringing the gas to temperature of the solvent.

The acetylene was bubbled through the absorption tube at the rate of about one bubble per second, in fact the gas was passed about as fast as is done in an ordinary combustion in the analytical laboratory.

Since volatile solvents are appreciably vaporized during this process of saturation, the exit gas and vapor in such case was passed through an ordinary potash bulb containing 95 per cent alcohol to catch the solvent which was later determined and proper connection made on the volume of solvent actually used. In 12 minutes the amount of solvent usually taken is completely saturated with the gas at atmospheric pressure (the pressure and temperature being always noted). The exit of the absorption tube was then connected to a similar tube two-thirds full of saturated calcium chloride solution which in turn had been saturated with acetylene. The calcium chloride was connected to an ordinary Hempel measuring burette (the liquid in the latter also being a saturated solution of calcium chloride subsequently saturated with acetylene).

The purpose of the calcium chloride was to absorb any solvent vapor that might be carried out in the solution of the gas, and which would otherwise be measured with the gas, giving too high a result. The saturated calcium chloride solution has a very low absorptive capacity for acetylene and it has been proved that it condenses and absorbs completely the vapors in each of the solvents tested. The efficiency of the saturated calcium chloride as an absorbent for the vapor of the various organic liquids suitable for acetylene solvents was demonstrated by boiling the solvents, and passing the vapor into such an absorbent tube, when the absorption was found to be complete. In several of the experiments noted below evolved gas from the measuring burette was bubbled back through a "potash" bulb containing 95 per cent alcohol, but no trace of solvent was found, which, if present, would have caused the gas reading to be too high.

This detail is mentioned here, because objection might be raised to the readings obtained with volatile solvents on the ground

that the gas would contain some of the vapor of the solvent, making a volume greater than the real volume of the gas.

The gas evolution begins soon after the absorption tube is removed from the freezing mixture. While the solvent was saturated at —18° C. usually, to guard against the possibility of the solvent not being saturated at the place taken as the starting point, the readings were not noted until the temperature of the solvent had risen to —10° C. The gas evolved from a known volume of solvent, saturated at —10° C. (since if gas is evolved between —18° and —10° it must be saturated at —10° C.), up to 30° C. is then measured, the figure obtained being recorded in each of the determinations noted below. The readings are given as actually obtained under the pressure and temperature conditions of the laboratory, and this gas volume is reduced to zero C. and 760 mm.

The reason for selecting —10° C. as a point at which the absorptions were determined, was that in commercial practice, it is very easy to cool the containers to this temperature. The 30° C. figure was obtained for the reason that with this the behavior of the solvent could be predicted in practical use, where the gas is rarely evolved at a temperature above 30° C.

Part II.—LABORATORY RESULTS.

The work of Claude and Hess (*Compt. Rend.* 124, 626) had shown that the absorption value of acetone far exceeded that of any solvent studied previously, and some preliminary skirmishing among organic liquids soon led to the view that the absorption of acetylene in acetone and other carbonyl compounds is partly chemical, in the sense that a chemical reaction or a partial reaction took place between the molecules of the absorbent or solvent and molecules of the acetylene. It is well known to organic chemists that the carbonyl group is a very reactive point in the molecule of many carbon compounds, in fact this indicates a condition of strain between the carbon and the oxygen, and the ready reactivity here is quoted in support of Baeyer's "Strain Theory."

Some French investigators, 10 or 12 years ago, working with higher acetylenes, actually obtained compounds which were addition products. The composition of the compounds that they

studied seemed to justify me in making the tentative statement, that there is a chemical action here, and I believe there is possibly a ring combination between the carbonyl carbon and oxygen and the acetylene, because of the unsaturated condition existing between the two carbon atoms of the acetylene.

With the conception then, that the carbonyl group is acetone was the reactive point, and that a chemical action of some kind took place, the solvents noted below were tested. The results established pretty clearly that there is a close relation here between the structure and the absorption capacity of a given compound for acetylene although the figures obtained for methylal and acetal would indicate that the "carbonyl theory" is not a complete enough one.

There is also a relation in a given series, usually, between the molecular weight and the absorptive capacity.

The figures obtained are arranged in the tables on pp. 136 and 137, the values determined for acetone and certain other organic liquids by previous observers being given for comparison.

Summary of Laboratory Results.

A study of the figures obtained establishes pretty conclusively that of all the liquids that have been tried, those organic compounds containing the carbonyl group are the best solvents for acetylene. We must exclude the organic acids, as the presence of the free hydroxyl hydrogen here seems to work counter to the chemical action upon which the remarkable solubility seems to depend. That the "carbonyl theory" is not satisfactory in every respect is shown by the high figures obtained for methylal and acetal. This peculiar action seems to require the assumption of quadrivalent oxygen for an adequate explanation.

Further, the figures clearly establish that in a given series the absorption of acetylene is greater the lower the molecular weight of the compound. The above experiments had in view the selection of a solvent that could be used industrially. Since the esters and acetals are out of the question industrially, requiring two and three molecular units per molecule of product, respectively, it was decided to try some larger scale experiments with acetaldehyde, making comparisons with other solvents in commercial use.

SOLUBILITY OF ACETYLENE ACCORDING TO PREVIOUS OBSERVERS

Solvent.	Temperature Degrees C.	Acetylene Dissolved by 1 Vol. Solvent.	Observer.
Acetone	15	25	Claude & Hess
Acetic acid	18	6	Berthelot
Alcohol	18	6	"
Benzoline (gasoline)	18	4	"
Chloroform	18	4	"
Paraffin oil	0	1	E. Miller
Paraffin oil	18	1.5	Berthelot
Carbon bisulphide	18	1	"
Olive oil	.....	0.48	Fuchs & Schiff
Carbon tetrachloride	0	0.25	Nieuwland

PRELIMINARY WORK ON SOLUBILITY OF ACETYLENE

(Figures refer to volume absorbed at −10 degrees C., but volumes are not reduced to standard conditions.)

Solvent.	Boiling-point of Solvent.	Acetylene Dissolved by 1 Vol. Solvent. at −10° C.	Remarks.
Ethyl mustard oil	131.5	3.2	
Ethylidine cyanhydrin	183	2.8	
Acetoacetone	137	10.2	
Benzophenone 1 g. dissolv. in 23 c.c. acetophenone	.....	.....	Saturated at 12° C., 1.7 vols. absorbed
Methyl propyl ketone	102	14.8	
Batyric aldehyde	74	10.3	
Acroelin	52	22.6	A crystalline compound of acetylene and acroelin forms during absorption
Propionaldehyde	48.9	24.2	
Acetaldehyde	21	54	
Acetaldehyde 50% by vol. Acetone 50% by vol.	36	42.2	
Acetaldehyde 50% by vol. Ethyl acetate 50% by vol.	43	40.2	
Acetaldehyde 50% by vol. Propionaldehyde 50% by vol.	32	31.1	

ABSORPTION FIGURES ON ACETYLENE SOLVENTS

(The averages in the last column represent absorption at −10° C. per c.c. solvent.)

Solvent.	Boiling-point of Solvent, C°.	Solvent Used. c.c.	Acetylene Evolved −10° to 30° C.c.c.	Acetylene Evolved −10° to b. st. of Solvent. c.c.	Acetylene Evolved per c.c. Solvent −10° to 30° C.c.c.	Acetylene Evolved per c.c. Solvent −10° to b.-pt. c.c.	Laboratory Temperature.	Barometer.	Corrected Volume Acetylene per c.c. Solv. −10 to 30°C.	Corrected Volume Acetylene per c.c. Solv. −10° to b.pt.	Average from Preceding Column.
Acetaldehyde C.P.	20.8	0.76	....	50.4	.....	66.3	23	745	.....	59.9	60.2
" "	20.8	0.81	....	52.6	.....	65.0	22	745	.....	58.8	
" "	20.8	1.44	....	98.8	.....	68.6	21.5	742	.....	62.1	
Methylal C.P.....	45.5	1	45.2	60.3	45.2	60.3	25.8	735	39.9	53.3	54.3
" "	45.5	1	45.4	62.4	45.4	62.4	25.4	735	40.1	55.2	
Acetal. C.P.....	103	1	20.4	30.7	20.4	30.7	18.8	742	18.7	28.8	28.8
Methyl formate C.P.....	32.3	1.5	70.8	81.5	47.2	54.3	25.6	742	42.1	48.5	48.4
Do.....	32.3	1.5	69.4	80.9	46.2	53.9	23.5	738	41.4	48.2	
Ethyl formate C.P.	54.5	1.5	46.1	71.1	30.7	47.4	24.5	738	27.4	42.3	42.2
" "	54.5	1.5	46	70.8	30.6	47.2	24.4	738	27.4	42	
Iso-amyl formate C.P.....	123	1.5	15.6	30.0	10.4	20	23.2	739	9.3	17.9	17.5
Do.....	123	1.5	15.9	28.7	10.6	19.1	23.8	739	9.5	17.1	
Methyl acetate C.P.....	57.5	1.5	61.3	82.8	40.8	55.2	22.2	735	36.6	49.5	52.3
Do.....	57.5	1.5	61.1	91.5	40.7	61	21.4	738	36.6	55	
Ethyl acetate C.P.	77	1.5	48.9	72.6	32.6	48.4	18.4	742	29.9	44.4	44.5
" "	77	1.5	48.4	71.8	32.3	47.9	17.2	742	29.6	44.6	
Iso-amyl acetate C.P.....	139	1.5	28.8	46	19.2	30.6	23.4	739	17.2	27.5	29.3
Do.....	139	1.5	29	51.7	19.3	34.5	21.8	738	17.4	31	

The amount of acetylene absorbed increases under pressure approximately according to Henry's Law, so the above laboratory results can be used to predict pretty closely what will be absorbed under the pressure used in the acetylene storage industry.

PART III.—LARGE SCALE EXPERIMENTS.

In this series acetaldehyde of between 99 and 99.5° purity was used as the solvent in a regular 6″ x 20″ acetylene storage tank, such as are commonly used on automobiles, in order to make comparisons with solvents in industrial use as to the amount of gas absorbed, the candle power of the light given on burning the

gas from the tank, the loss of solvent, etc. The other solvents were C. P. Acetone, and a complex mixture of organic liquids, which is used as a solvent for acetylene, and which will be referred to in the accompanying curves as Ester-Ketone-Aldehyde solvent, since it undoubtedly owes its absorbent power to the presence of bodies belonging to these three groups.

Probably the most important point of comparison is brought out in the curve for each solvent where the candle power at various times of the discharge is shown; a striking difference between the volatile and the non-volatile solvents appears here. With the non-volatile solvents there is little more than an hour's warning before the gas is gone completely, while with the volatile acetaldehyde solvent there is an interval of from 4 to 6 hours in length from the first warning and the "going out" of the light. With the acetaldehyde, there is a round black spot in the flame that makes its appearance at about the 35 candle power point on the curve, and the size of this spot increases as the candle power drops, its appearance giving about 6 hours warning, where two ½ cu. ft. burners are being used.

The loss of solvent, which runs with the non-volatile solvents is common practice from 4 to 6 ozs. for each discharge of the tank, was a fraction over 12 ozs. in the acetaldehyde experiment, where the evolution of the gas was pushed to the limit, and would undoubtedly run about 8 ozs. in industrial use.

At first glance it appears rather surprising that the drop in candle power with the increase of solvent vapor in the gas, is not greater. For example, it is seen from the curve where candle power is plotted against per cent of solvent vapor in the gas, that when the solvent vapor has increased to 80 per cent, the candle power is still above 20. It has been noted by other observers that diluents lower the candle power of acetylene more rapidly the lower the flame temperature of the diluent. Since acetylene has a heat of 313.8 cals. per gram molecule, and acetaldehyde has 279.2, we have a satisfactory explanation of the action of the diluent in this case; the calculated temperature of the hottest part of the oxy-acetylene flame is in the neighborhood of 4000° C. and that of the oxy-acetaldehyde flame is above 3400° C.

The aldehyde vapor is a good diluent also for the reason that the volume of air or of oxygen required for its combustion

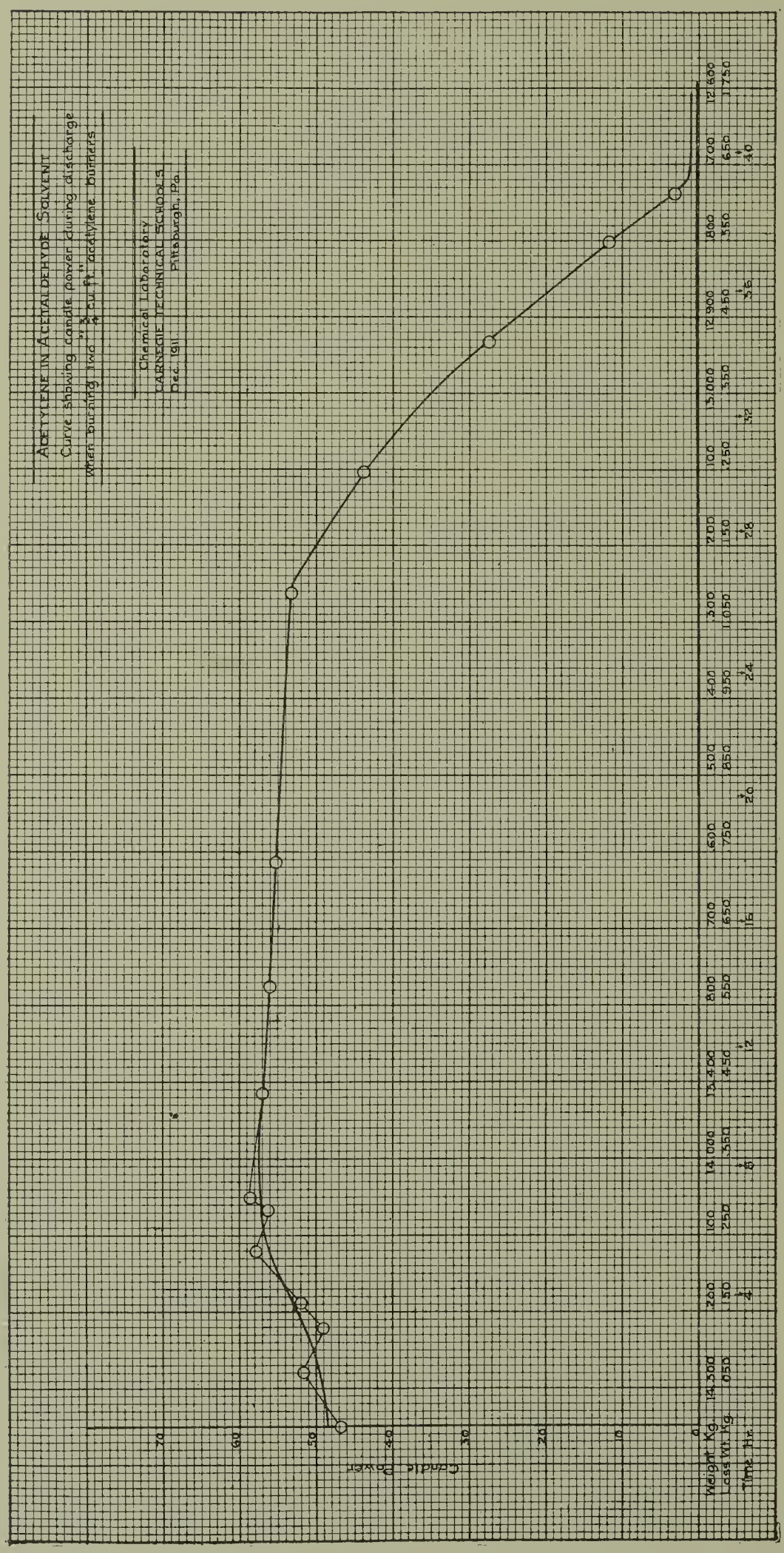


PLATE I.—Acetylene in Acetaldehyde Solvent.
Curve showing candle power discharge when burning two "¾ cu. ft." acetylene burners.

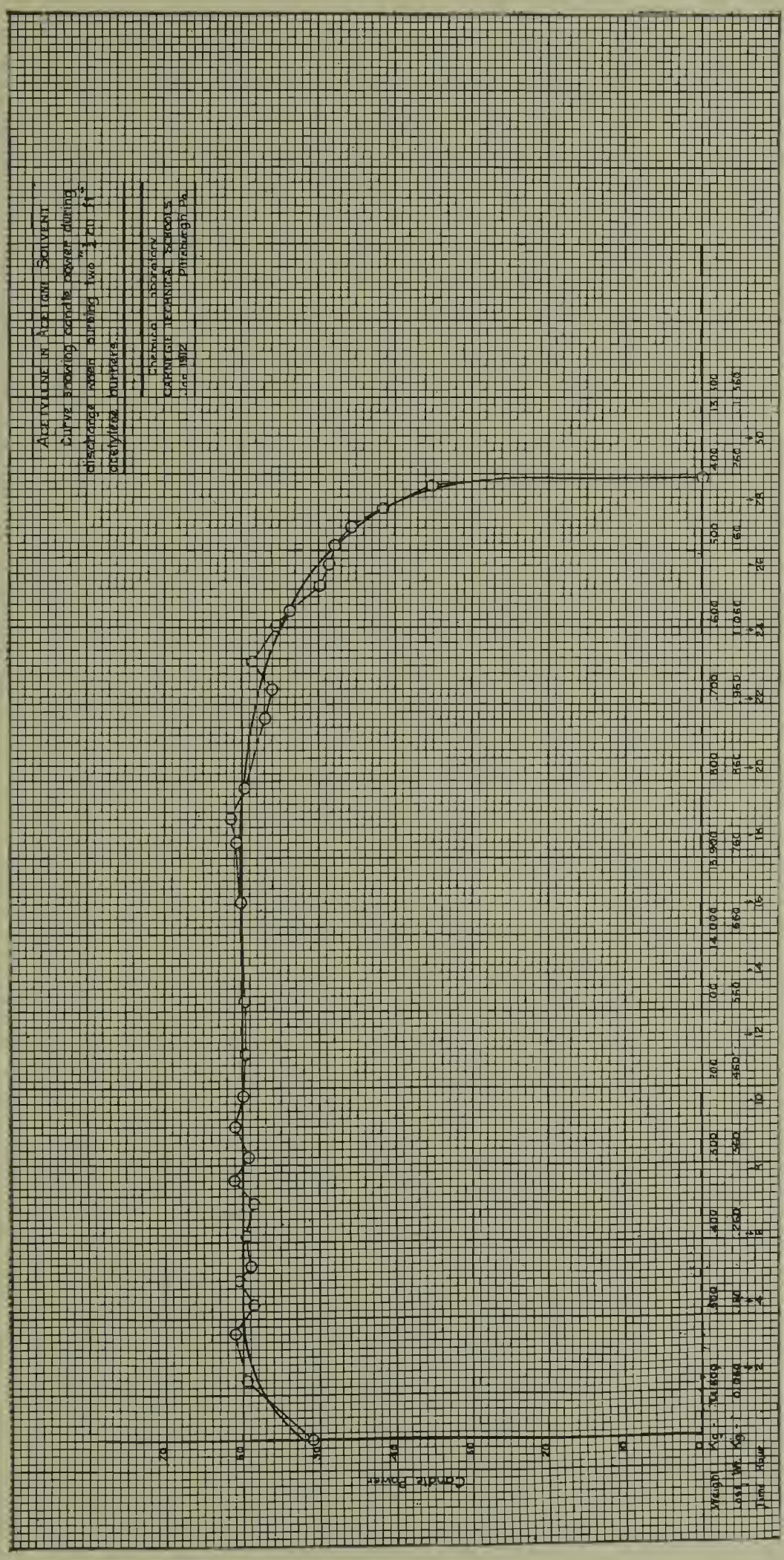


PLATE II.—Acetylene in Acetone Solvent.
Curve showing candle power during discharge when burning two "¾ cu. ft." acetylene burners.

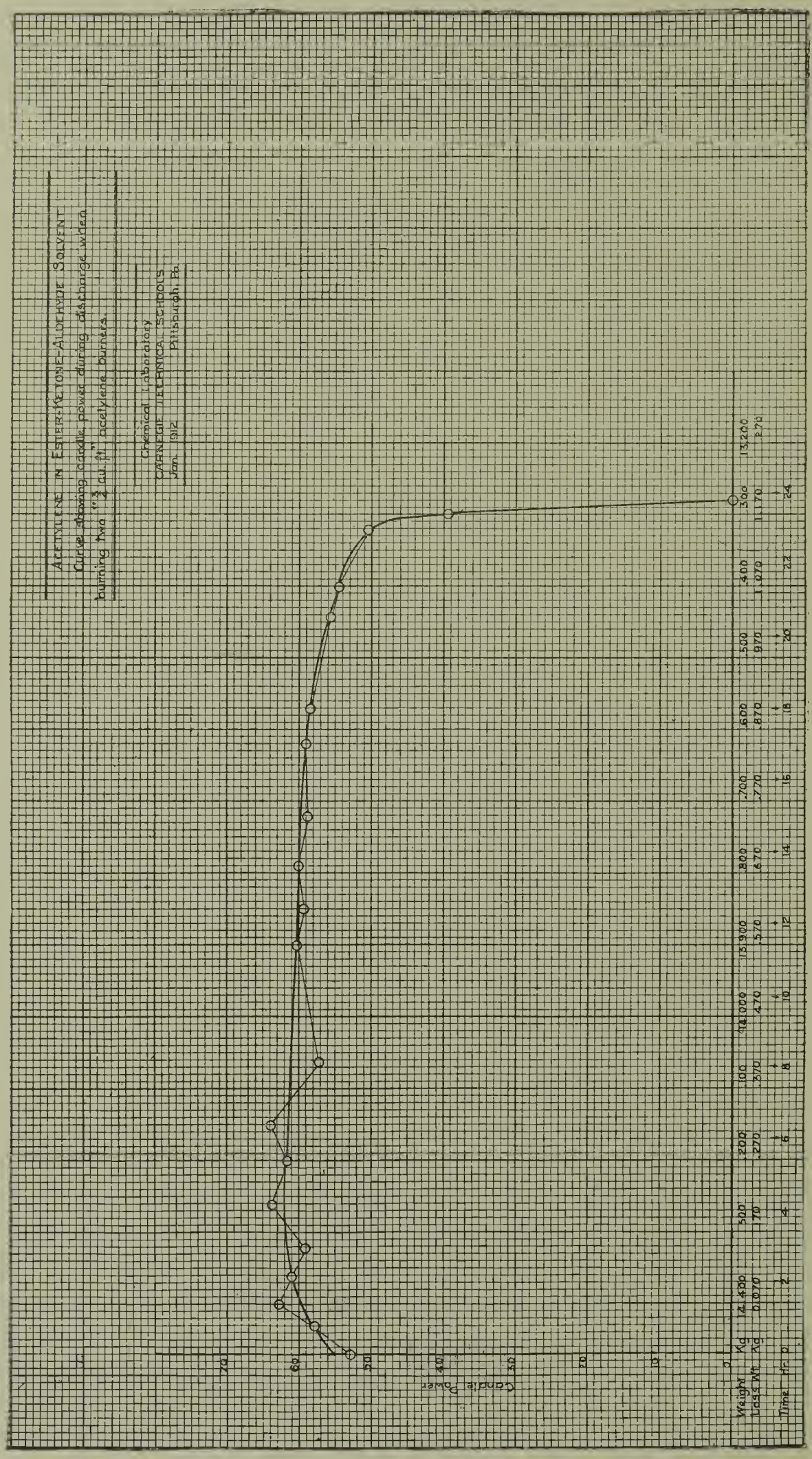


PLATE III.—Acetylene in Ester-Ketone-Aldehyde Solvent.
Curve showing candle power during discharge when burning two "¾ cu. ft." acetylene burners.

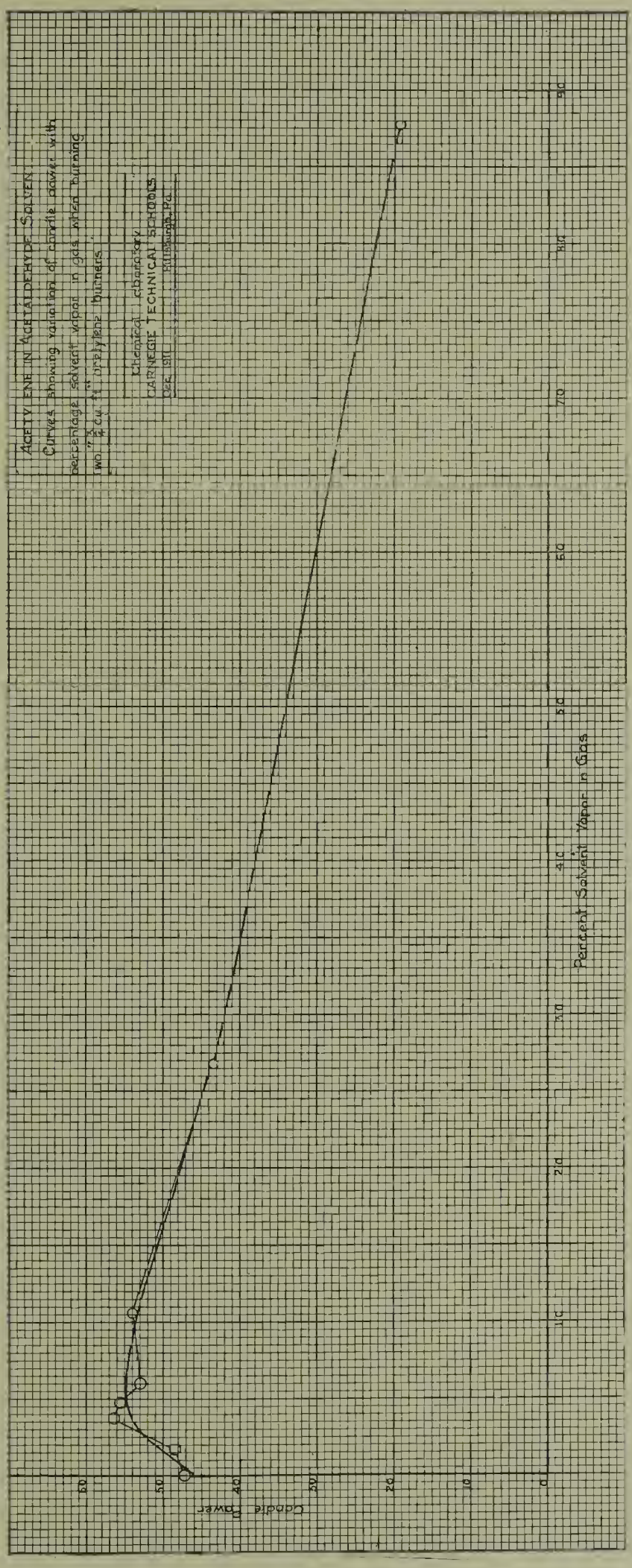


PLATE IV.—Acetylene in Acetaldehyde Solvent.

Curve showing variation of candle power with percentage solvent vapor in gas when burning two "¾ cu. ft." acetylene burners.

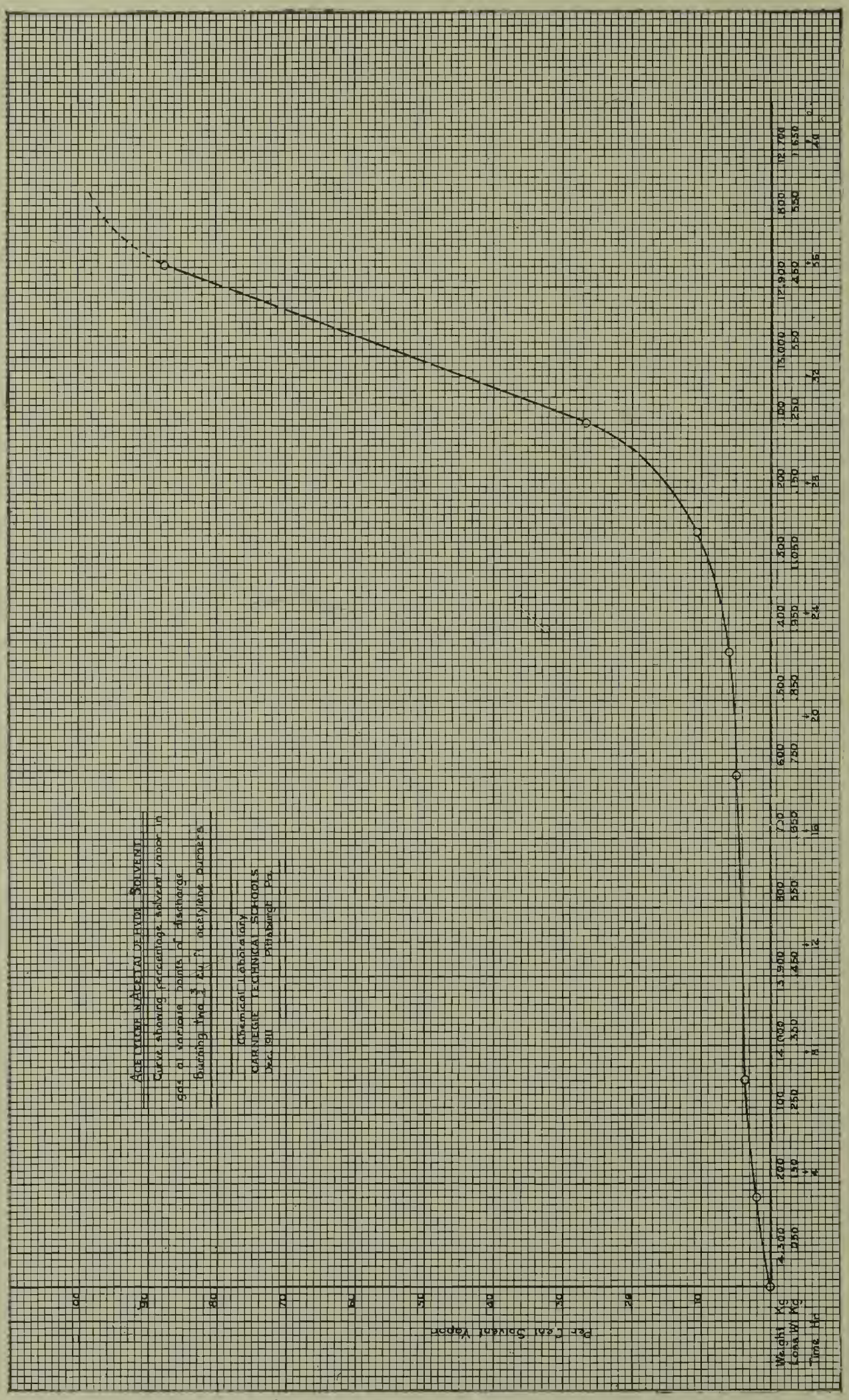


PLATE V.—Acetylene in Acetaldehyde Solvent.

Curve showing percentage solvent vapor in gas at various points of discharge. Burning two "¾ cu. ft." acetylene burners.

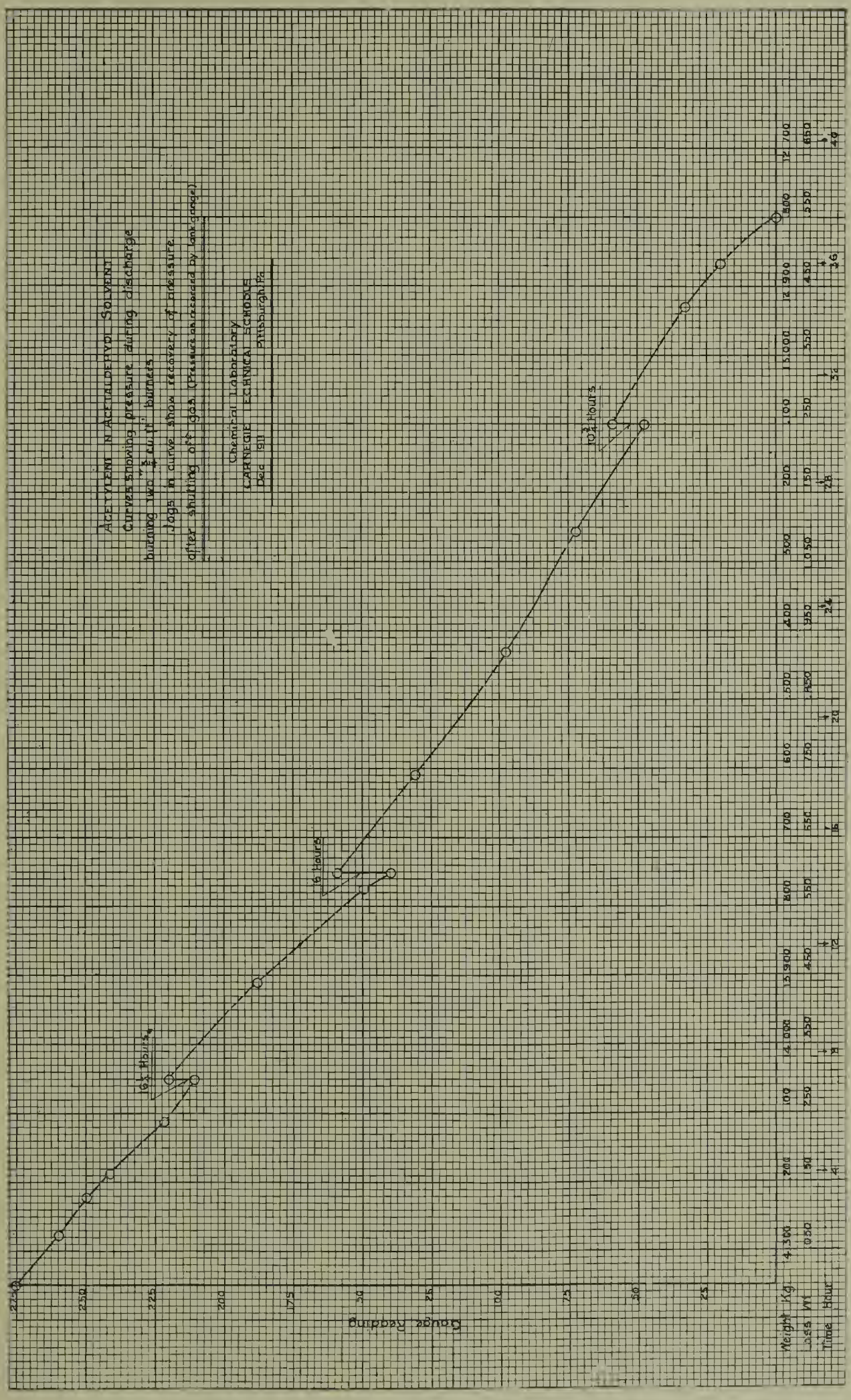


PLATE VI.—Acetylene in Acetaldehyde Solvent.

Curve showing pressure during discharge burning two "¾ cu. ft." burners.

Jogs in curve show recovery of pressure after shutting off gas (pressure as recorded by tank gauge).

is theoretically exactly the same as that required for acetylene, so that there is scarcely any change in the shape of the flame, as the percentage of aldehyde vapor increases.

These two facts, the high heat of combustion, and the equality of the volumes of air or of oxygen needed make for an advantage in the use of acetaldehyde as a solvent for acetylene to be used in welding and cutting operations. In an emergency repair job in a remote locality, in case the gas gives out the work can be finished by drawing on the volatile solvent for the combustible.

Long observation has shown that the figures attained in industrial practice with the non-volatile solvents noted above average 37 ozs. acetylene in 85 ozs. solvent, at a temperature of 70° F. and a pressure of 250 lbs. gauge. In this experiment with acetaldehyde as the solvent, 48 ozs. of acetylene was absorbed in 82 ozs. of solvent, with the gauge standing at 265 when the temperature rose to 70° F. This figure shows that acetaldehyde is a liquid that has a superior absorbent power for acetylene, in fact the author ventures the statement that this experiment shows an amount of acetylene greater than has ever before been stored in a given volume of solvent.

Conclusions.

The rapidly advancing price of acetone and other solvents makes it desirable to have commercially available a solvent that can be obtained in any quantity and which shall not advance in price abnormally.

Acetaldehyde as can be seen from the foregoing experiments, fulfills the industrial requirements; its volatility can actually be turned to advantage, as noted above.

Since acetaldehyde can be made in one chemical operation directly from denatured alcohol, we have here a source of supply of an acetylene solvent which will not increase in price, but which will undoubtedly become cheaper as improved methods of agriculture make it possible to produce denatured alcohol cheaper.

Acknowledgment.

I wish to state that I am indebted to my former student assistants, Messrs. E. P. Poste and E. W. Gardner for their help in taking readings and making records in the above experiments. In

this connection, I wish to express my thanks to Dr. H. S. Hower of the Physics Department, Carnegie Institute of Technology, for assistance in taking the candle power readings and for the loan and standardization of the Brodhun Portable Photometer, which was used in the photometric part of the work.

Chemical Department,
Carnegie Institute of Technology.

December, 1912.

DISCUSSION.

PRESIDENT: Gentlemen, this is a very suggestive paper, because it has a direct bearing upon the important subject of storing and using acetylene generally. Not only for purposes of illumination, but also for acetylene welding. When I was a university student I remember very well that acetaldehyde was cited in research work as one of the expensive luxuries. It was sold then for something like a hundred dollars a kilo, but before I graduated, some German alcohol manufacturers by their methods of distillation began to produce aldehyde as a by-product and soon it was possible to buy aldehyde for a few marks a kilo. It is possible that these gentlemen are able to produce acetaldehyde in large quantities, and that it will become a real commercial commodity, so that the use of acetaldehyde will develop; it is the same old story again, to bring a commodity in, supply it, and right away it may develop that if acetaldehyde is thrown into the market for acetylene that they may use it for a lot of other purposes, and I understand that attempts are being made to utilize it as a solvent. It seems to be destined to become a competitor of acetone. Acetone has been increasing in price all the time, and is, after all, a by-product of wood distillation; it is therefore of limited production, unless those fermentation methods of production which lately have been announced in England and France give us a cheaper supply of acetone, but we have not heard much since that first announcement, which was made about three months ago.

The subject is now open for discussion.

Prof. BAIN: I have learned a lesson to-night in a very curious way. I walked into the laboratory of one of my colleagues last winter and this gentleman is very much interested in organic

chemistry and is devoting all his time and attention to it, and I cannot say that my ideas run in the same direction. When I walked in and saw him pouring something into a large flask I asked him what it was, and he said he was testing the action of acetylene on acetone. I asked him how that came about, and he said he had been consulted by an agent who was compressing acetylene in the cylinders for the railway companies, and he got interested in it, and he warmed up to the subject and told me a whole lot about it, but, much to my sorrow, I have to say that I turned a rather deaf ear to it. That man knew what he was talking about, and now I am trying to think what he told me. He told me, as far as I can remember, that there is formed a series of compounds by the action of acetylene on the compound, and the only substance I can remember is that phorone is one of the compounds formed there, and there are a number of others. I am under the impression that he has never published the results, but I know he spent a good deal of time on it, and no doubt before very long he will be able to know exactly what compounds are formed in its reaction.

Dr. ITTNER: I would like to ask if Mr. James has made experiments on the stability of acetaldehyde itself, whether it changes readily or not, or whether it is sufficiently stable. Of course acetone is under ordinary conditions a very stable compound, but acetaldehyde—does it answer the same qualifications and is it stable?

Prof. JAMES: No, it is not as stable, and one thing we have to avoid in its use is the presence of alkalis, or the presence of acids. It is necessary to be very careful about the containers. They are filled with a porous filling, and in the old method of preparing that filling, sodium silicate was used, and we have to avoid that. In answer to Dr. Bain, I venture to suggest that his friend is mistaken. The acetylene can be all driven out of any solvent by boiling. My opinion is that this is a chemical action, and the formation of some compound which decomposes as soon as the pressure is released.

Prof. BAIN: I simply said that it was my neglect and I paid no attention to it at the time, so I will not be able to offer you any information except that simple statement.

PRESIDENT: There is one possibility about acetaldehyde. Its

tendency to produce acetic acid by oxidation. Of course it is only possible in the presence of air. If your acetylene contains air and you compress it, it will produce acetic acid in the steel containers.

MEMBER: I think Prof. James is to be congratulated. One of our fellow members in this country has started on a large scale in making acetaldehyde.

MEMBER: May I ask if acetaldehyde is on the market now?

Prof. JAMES: No, it is not. We sent out samples to find out if there should be a demand for it in other lines. We propose to use it entirely for this industry (acetylene storage) and incidentally, if we find a field outside we will make more of it. Of course we would have to ship it in the form of 50 per cent solution in methyl alcohol. It is a pretty good solvent, about as good as amyl acetate, for the nitrocelluloses for example, and we hope to introduce it for that purpose, but we have not as yet.

MEMBER: It might be interesting to remark that a proposal has been taken for the use of butylidine glycol instead of glycerine. It is calculated that if the price of glycerine should become about 24 cents a pound, that butylidine glycol would do for a substitute in explosive manufacture.

THE NEW CHEMICAL ENGINEERING COURSE AND LABORATORIES AT COLUMBIA UNIVERSITY

By M. C. WHITAKER.

Read at the Detroit Meeting December 6, 1912.

Course of Instruction.

Improved training in Chemical Engineering is a problem which has received much consideration from this Institute. The Committee on Education has done valuable work in collecting data and opinions from leading chemical engineers and has offered many helpful suggestions on this important subject. While no specific curriculum has been officially adopted and no definite system of laboratory equipment or training has been agreed upon, it is felt that certain points stand out with sufficient prominence to justify action, and it is upon this basis that we have gone ahead and initiated the work as here outlined.

The burden of the rapid advance in all engineering science has been felt more keenly by the student than by anyone else. The amount of ground to be covered between high school graduation and the engineering diploma has increased year by year, but the time allotted for the work has remained the same,—four years. When the student's "elastic limit" is reached it becomes necessary to curtail at some point. In some of the schools cultural subjects are being eliminated from the curriculum to the great loss of the student's general scholarship; in other institutions fundamentals are superficialized to such an extent that the graduate lacks the necessary foundation on which to develop; in still other engineering courses, cultural and fundamental subjects are retained and the engineering applications are given absent treatment. In a few cases, where the faculty is about evenly divided between cultural and engineering representatives, the student's wail concerning the

amount of time at his disposal for study, and the assimilation of the various and varied subjects offered in the distended curriculum, has been drowned by the rhetorical scramble for more time for each of the "most important subjects" in the course.

The obvious remedy to meet this deplorable situation and to provide for the rapidly advancing demands of technical education is to give the student more time to do the increased amount of work.

The engineering departments of Columbia University will become, in 1914, regular post-graduate professional schools and require a college degree or equivalent training for admission. It has been noted for several years that over 20 per cent of the students in our engineering departments possessed the college degree at entrance.

The college training taken as a preliminary preparation to this post-graduate course in engineering must necessarily include the fundamental mathematics, physics and chemistry, in addition to the usual college courses. In a carefully arranged curriculum this ground may be covered in three years. I submit herewith the course planned by the faculty of Columbia College to meet the requirements for admission to the new post-graduate engineering schools. The subjects treated in this undergraduate course are now to be found in the schedules of practically all colleges and by judicious elections, the required fundamentals may be satisfactorily completed in any good institution.

A THREE YEAR COURSE OFFERED BY COLUMBIA COLLEGE TO FULFILL REQUIREMENTS FOR ENTRANCE TO POST-GRADUATE ENGINEERING SCHOOLS.

FIRST YEAR

FIRST HALF		SECOND HALF	
Advanced Algebra (Math. 1)	2	Analytical Geometry (Math. 4)	3
Chemistry, Gen'l (2 Lect.) (Chem. 3c.) (6 Lab.)	5	Chemistry, Gen'l (2 Lect.) (Chem. 4c.) (6 Lab.)	5
English Composition (Engl. A)	3	English Composition (Engl. A)	3
Principles of Science: (Philosophy A)	3	Principles of Science: (Philosophy A)	3
Modern Language based on Intermediate Entr. requirement	3	Modern Language: (*continued*)	3
Shop Work (1 Aft.)	1	Shop Work (1 Aft.)	1
Physical Education A	1	Physical Education A	1
		Total Points	37

SECOND YEAR

FIRST HALF		SECOND HALF	
Calculus (Math. 15)	3	Calculus (Math. 16)	3
Chemistry (Chem. 67) (2 Lect.) Qualitative (6 Lab.)	5	Chemistry (Chem. 68) (2 Lect.) Qualitative (6 Lab.)	5
Introduction to Eng. Lit.: (English B 3)	3	Physics (3 Lect., 3 Rec.)	6
Epochs of History (Hist. A)	3	Epochs of History (Hist. A)	3
Drafting (2 afternoons)	2	Drafting (2 afternoons)	2
Geometry: Descriptive (Drafting 3)	3	Total points	38

THIRD YEAR

FIRST HALF		SECOND HALF	
Calculus (Math. 17)	3	Calculus	3
Physics (3 Lect.,) (3 Rec. 1 afternoon Lab.)	7½	Physics (same)	7½
Political Science (Econ. 1)	3	Statics (Mechanics 2)	3
Mineralogy (2 Lect.) (1 afternoon)	3	Political Science (Politics 4)	3
		Surveying (Civil Eng. 2)	2
		Total points	35

To make up the requirement of 124 points for the degree of B. S., 14 additional points must be made either by extra credit for high standing, by work in Summer Sessions or by free election during the second and third year of the program; such election may be made at the student's option from among the courses in which he can satisfy the prerequisites, with the single provision that no program aggregating more than 22½ points will be approved for any half-year.

NEW THREE YEAR POST-GRADUATE COURSE IN CHEMICAL ENGINEERING.

FIRST YEAR

FIRST HALF			SECOND HALF		
Subject	*Hrs.*	*Aft.*	*Subject*	*Hrs.*	*Aft.*
Physical Laboratory	0	1	Advanced Heat	3	0
Mechanics	3	0	Mechanics	3	0
Industrial Chemistry	3	0	Industrial Chemistry	3	0
Power Mahcinery	2	0	Power Machinery	2	0
Physical Chemistry	3	1	Physical Chemistry	3	1
Elements of Electrical Engineering	2	0	Electrical Machinery	2	0
Hydraulics—Theory	2	0	Hydraulics—Laboratory	0	1
Quantitative Analysis	2	3	Quantitative and Engineeirng Chemistry	2	3
Total	17	5	Total	18	5

SUMMER WORK.

CHEMICAL FACTORY INSPECTION (2 WEEKS)

FACTORY WORK AND DETAILED REPORT ON SOME ASSIGNED INDUSTRY (6 WEEKS)

SECOND YEAR

FIRST HALF

Subject	*Hrs.*	*Aft.*
Organic Chemistry	3	2
Machine Elements	2	0
Food and Sanitary Chemistry	3	0
Engineering Thermodynamics	3	0
Direct Current Laboratory	1	1
Resistance of Materials	5	2
Total	17	5

SECOND HALF

Subject	*Hrs.*	*Aft.*
Organic Chemistry	3	2
Machine Elements	2	0
Adv. Industrial Chemistry	3	0
Engineering Thermodynamics	5	0
Alternating Current Lab	1	1
Assaying	2	2
Total	16	5

SUMMER WORK.

CHEMICAL AND MECHANICAL ENGINEERING LABORATORY (8 WEEKS)

THIRD YEAR

FIRST HALF

Subject	*Hrs.*	*Aft.*
Introduction to Metallurgy and Metallurgy of Copper	3	0
Metallurgy—Iron and Steel	1	0
Chemical Factory Machinery	3	0
Electrochemistry	2	1
Gas Power	2	1
Commercial Organic Analysis	2	3
Seminar	5	0
Total	18	5

SECOND HALF

Subject.	*Hrs.*	*Aft.*
Metallurgy—Lead, Zinc, Gold, and Silver	3	0
Chemical Factory Management	3	0
Steam Power	4	2
Business Law	2	0
Chemical Engineering Lab.: Special Problems	0	3
Seminar	6	0
Total	18	5

Corresponding courses have been adopted for Mining, Civil, Electrical and Mechanical Engineering and it is believed that this extension of practically two years in the amount of time to be devoted to the fundamental and professional work together, will not only relieve the present tension on instructors and students, but will produce a class of graduates immeasurably better qualified to assume the responsibilities of their profession.

CHEMICAL ENGINEERING LABORATORY.

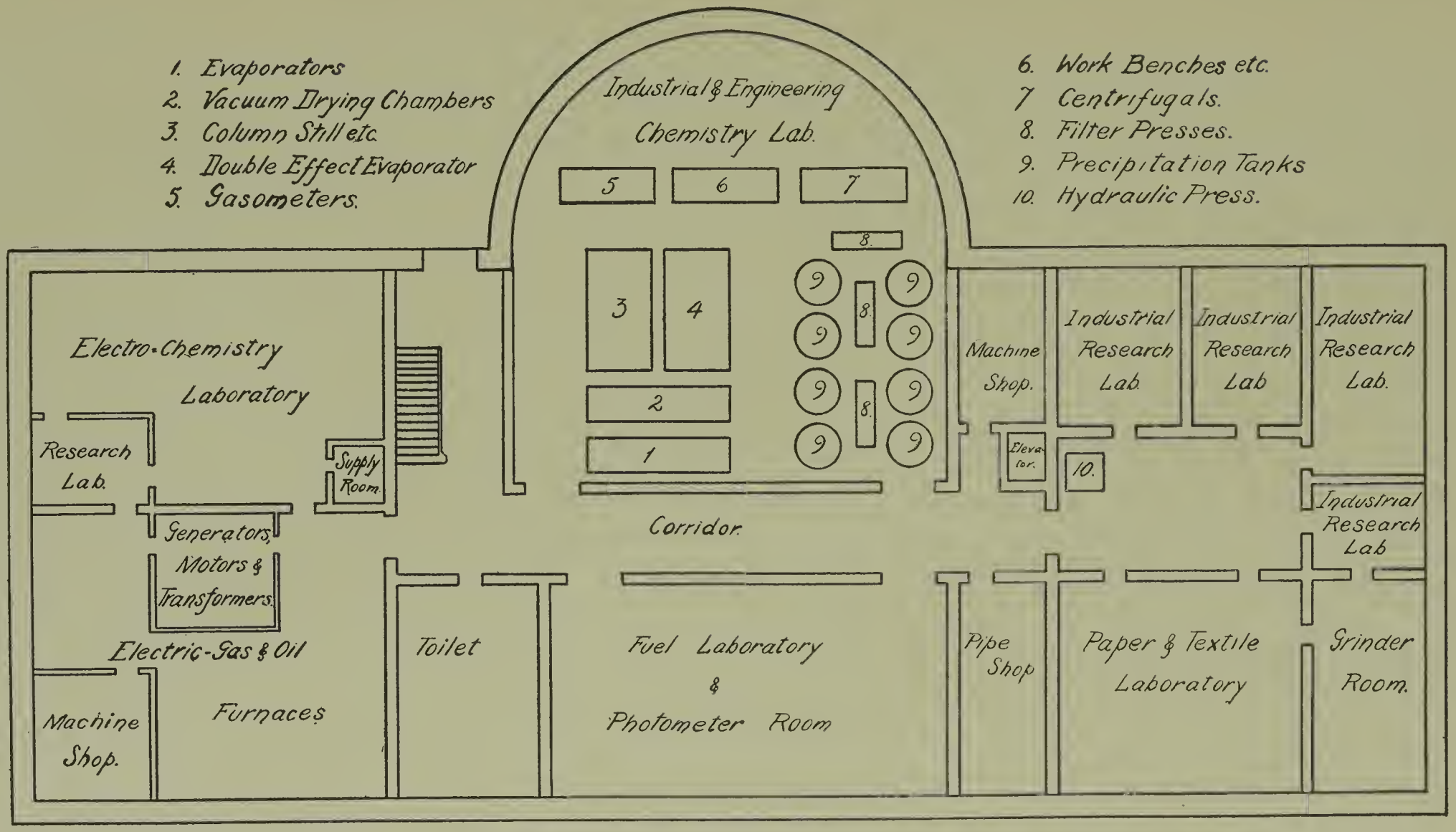
Chemical Engineering courses must eventually teach men to use engineering methods and engineering appliances in the solution of chemical problems and the operation of chemical processes. These engineering methods and appliances are not to be found

in the test tube, beaker, or funnel of the chemical laboratory any more than steam engines, hydraulic machinery, electric generators, and switch boards are to be found in a physics laboratory. Mechanical and electrical engineers long since saw the difference between the equipment needed for studying the application of the fundamental scientific principles to complicated engineering conditions, and the equipment to be used to study the laws and principles upon which these fundamentals are based, and accordingly established laboratories equipped with mechanical and electrical engineering appliances to meet these needs.

The chemist, on the other hand, with the characteristic conservatism produced by many disappointments, is slow to recognize the point at which the study of principles ends and the study of applications begins. He hesitates to develop laboratories with tanks, siphons, pumps, filter presses, evaporators, stills, centrifugals, absorption towers, etc., but prefers to consider his work complete with the establishment of a principle on a test tube scale. The chemical engineer is sorely needed at this point to take chemical principles and engineer them just as the mechanical engineer engineers the physics of heat, or the electrical engineer engineers the physics of electricity. The chemical engineer has small chance of engineering chemical operations unless he knows the fundamental methods and appliances available. He cannot establish data with a beaker and a test tube on which to engineer a process any more than a mechanical engineer can arrive at a correct conclusion in regard to the performance of a steam boiler by some experiments with a tomato can.

It seems clear to me therefore that our students must study the methods, appliances and engineering principles involved in chemical operations by contact with the equipment developed for this field. Furthermore, our researches, where industrial application is sought, must be transferred from the beaker to the tank, from the funnel to the filter press, from the evaporating dish to the vacuum pan, from the distilling flask to the still, and so on, before any data on which to base judgment as to its practicability can ever be established.

I am establishing at Columbia, with the support of Dean Goetze and an administration which seems to have the courage of my convictions, a chemical engineering laboratory in accordance with



→ Department of Industrial Chemistry ←

First Floor Plan

Havemeyer Hall — Columbia University

FIG. 1.

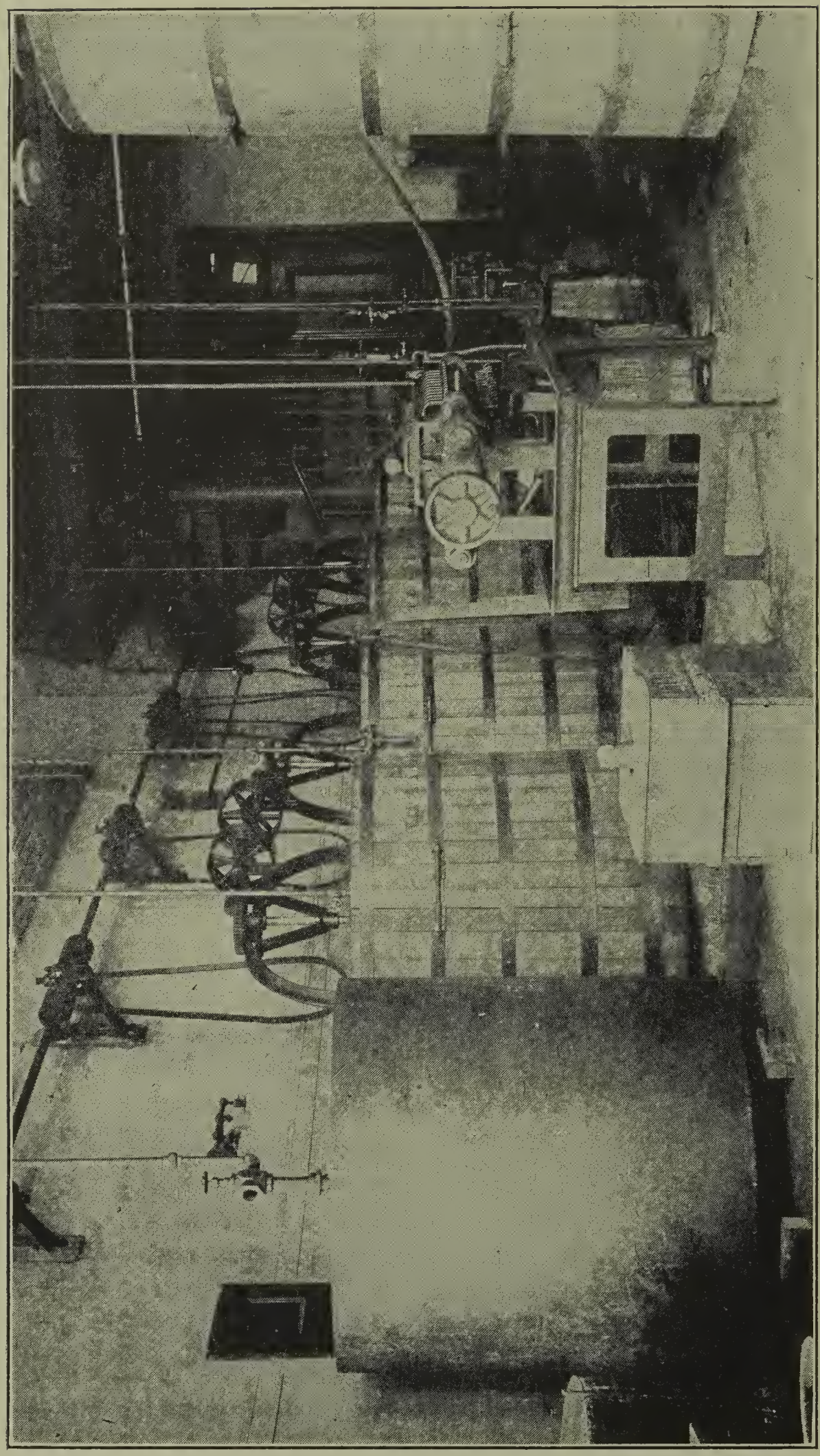


FIG. 2.—Tanks, Filter Presses, Agitators, Etc.

the plan outlined. The accommodations are by no means ideal and the scheme is far from complete, but we have made a bold

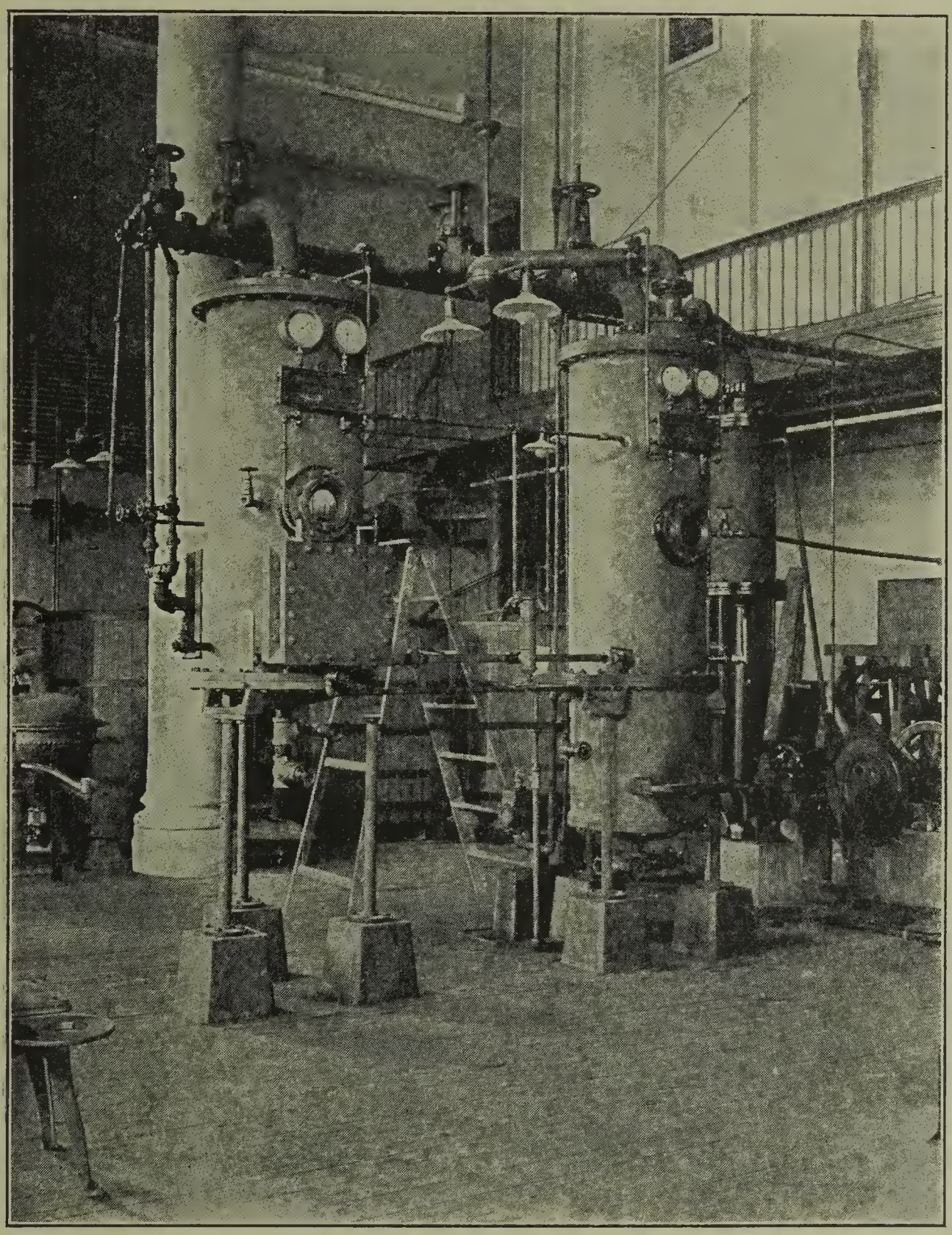


FIG. 3.—Double Effect Vacuum Pans.

beginning. We hope to expand rapidly from year to year and ultimately have a laboratory of chemical engineering comparable with the best laboratories in mechanical and electrical engineering.

Some photographs will serve to show the plan and scope of our work and the progress thus far made. The chemical engineer-

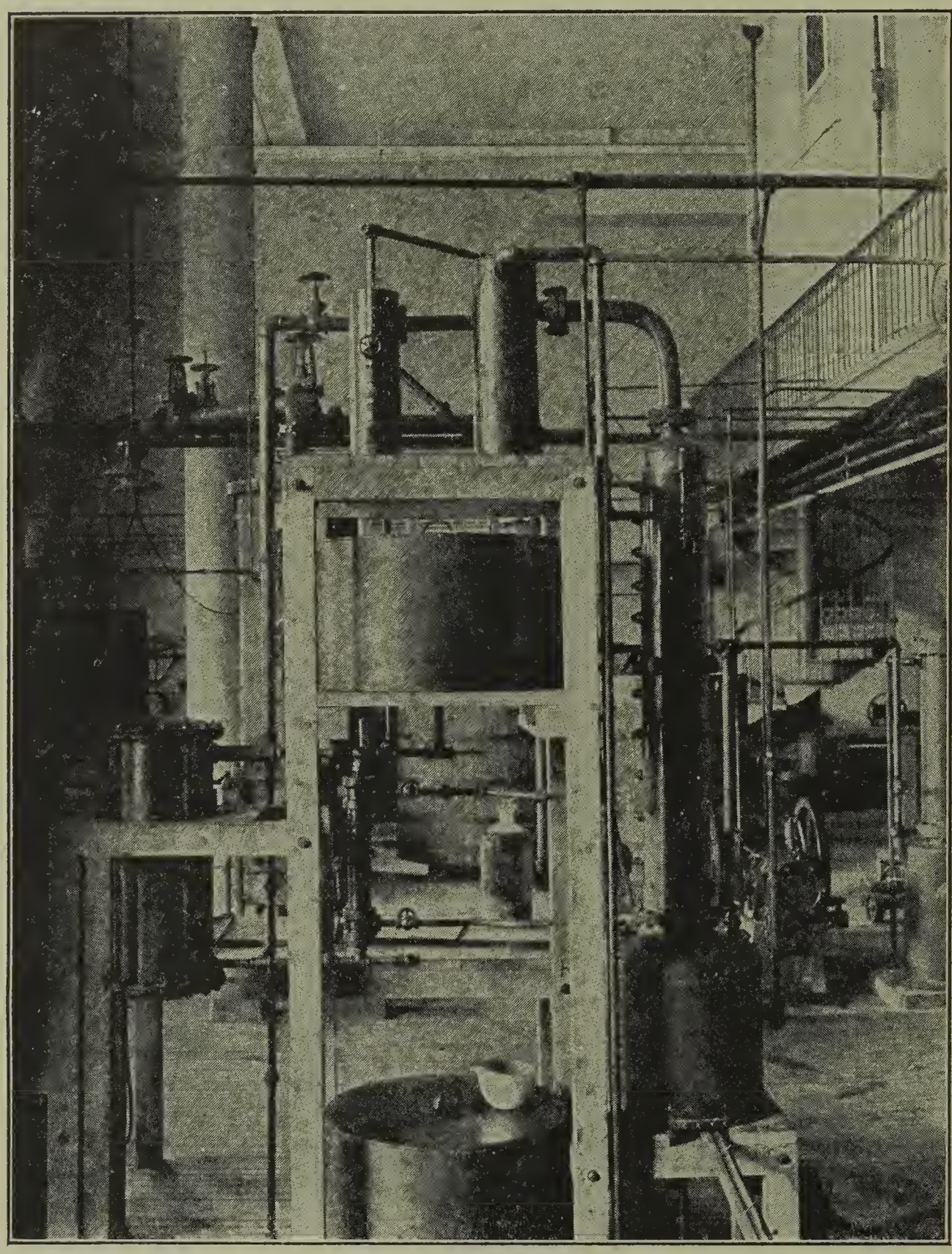


FIG. 4.—Column Still and Extractor.

ing laboratories and the electrochemical laboratories occupy the entire lower floors of the Havemeyer Building. The division of space is shown by reference to Fig. 1, from which it will be noted

that we have, besides the large general laboratory, the electrochemical laboratory, paper and textile laboratory, research laboratories, grinder room, pipe shop, machine shop, etc. Some of the

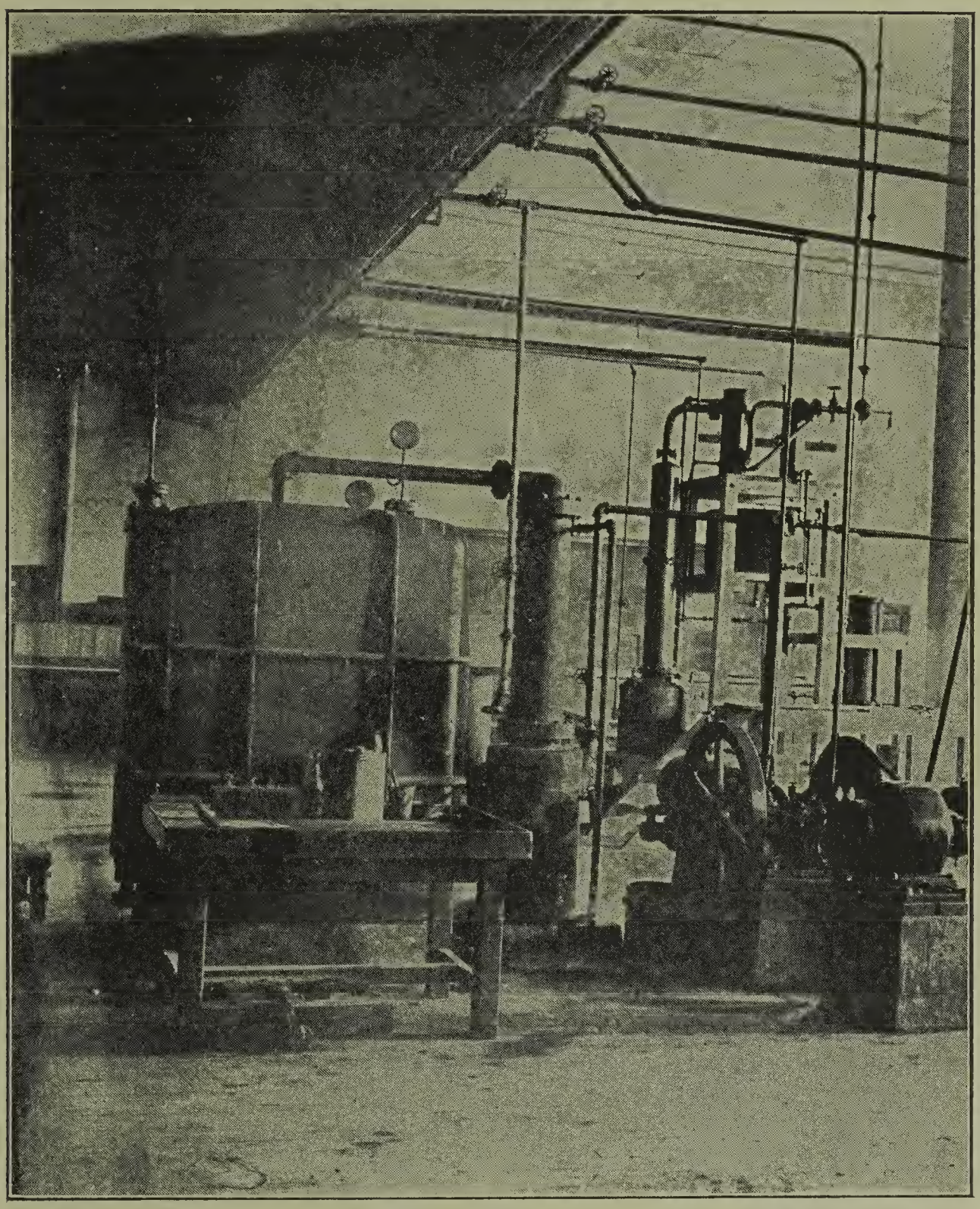


FIG. 5.—Shelf Drier and Vacuum Pumps.

more typical equipment now installed and in operation will be illustrated by reference to Figs. 2 to 7.

The Chemical Engineering students use this apparatus not

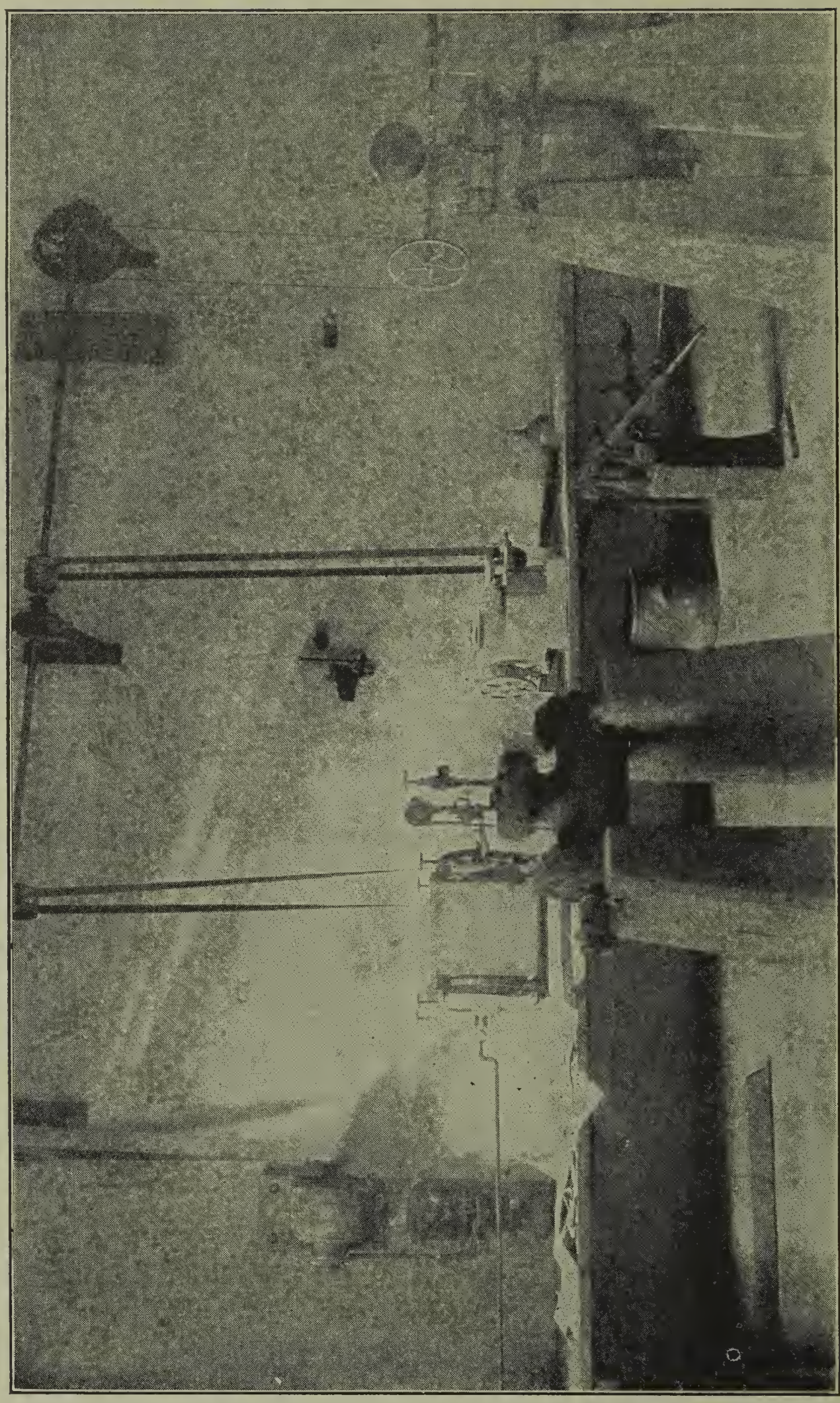


FIG. 6.—Paper Laboratory.

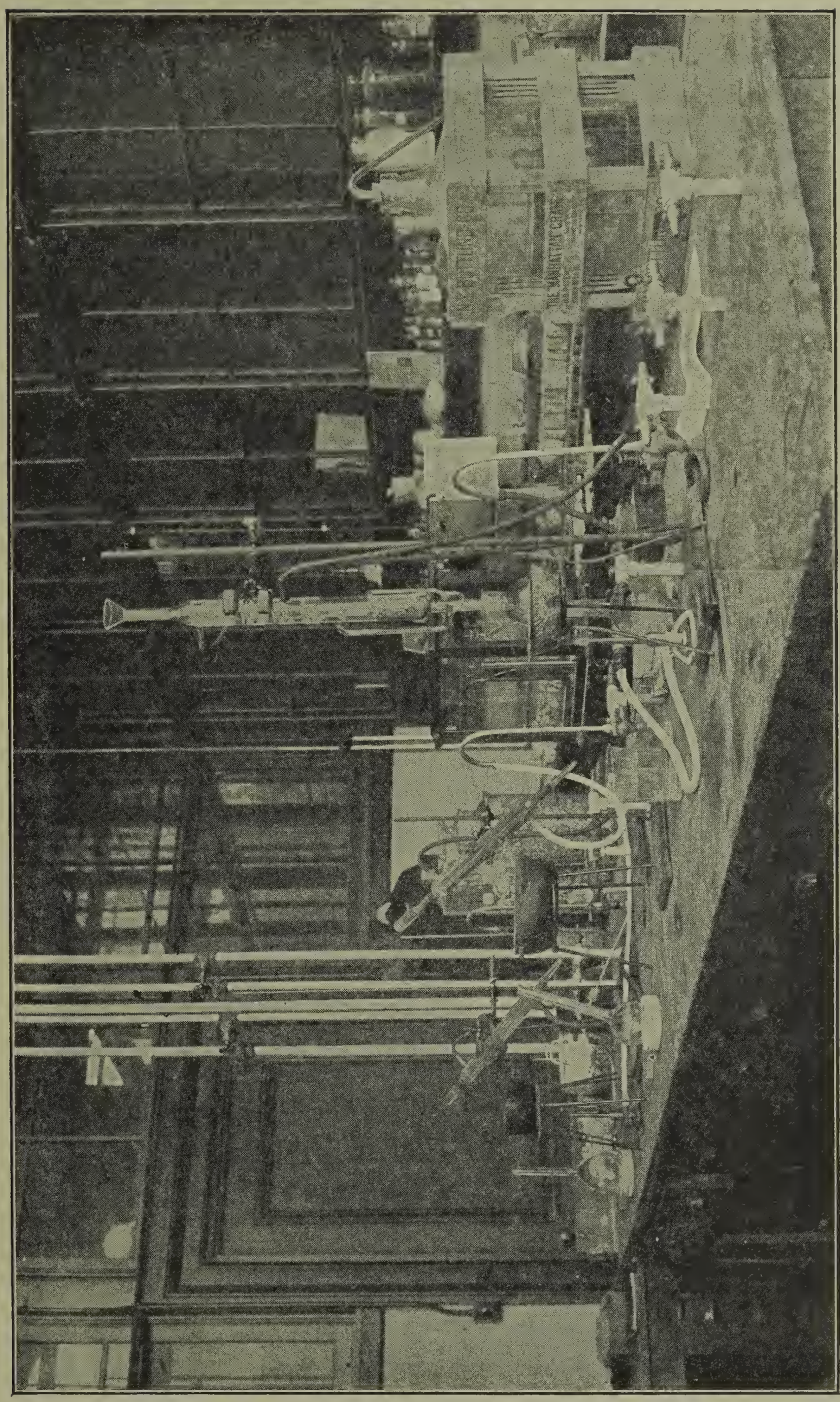


FIG. 7.—Laboratory for Industrial Research.

only as types of equipment already developed to carry out the desired operations, but they soon become familiar with the limitations of the appliances and have to exercise engineering judgment in overcoming the difficulties. The research men find standard equipment of modern and approved type ready and available for trying out any fundamental practice on a scale and in a way which will enable them to establish data capable of being used in business calculations or works engineering. The problems arising as a result of transferring operations from the laboratory to the large scale factory appliance may here be met and solved under the sympathetic eye and patient hand of the developer, instead of being doomed to failure by being delivered in incomplete form to unsympathetic and busy works managers.

The problems assigned in all of these operations have been selected so as to be cyclic, and thus avoid production operations. For example, in the work involving the use of the tanks, siphons, pumps, filter presses, etc., a squad of four or five students begins by dissolving a weighed quantity of the waste sulphates of the didymium earths in dilute sulphuric acid in a lead lined tank with air agitation. This solution is transferred with a lead siphon and precipitated as an oxalate, the liquid returned to the lead tank for future use, the precipitate washed by decantation, filter-pressed, the press cake transferred to an iron tank and converted to hydroxides. The soluble oxalate is stored for use in the evaporator and the hydroxides are dissolved in acid and returned to the first tank. During these operations, the student has had experience with solution, precipitation, washing by decantation, filtration of a granular crystalline precipitate, conversion from a solid insoluble in acid to one soluble, filtration of a slime and resolution in acid, air, steam and mechanical agitation, pumps, siphons, etc. All of this work is done on a quantitative basis and losses are checked and accounted for at each stage of the work.

Similar cyclic operations have been devised for all of the units of the laboratory with the object of reducing operating expenses to a minimum and also avoiding the accumulation of a product. Students are given the greatest possible liberty in the methods of handling their problem, are allowed to make mistakes and get experience. The instructors are there to assign problems and supervise the work, but not to dictate and direct.

DISCUSSION.

MEMBER: I would like to ask Prof. Whitaker in just which course on this schedule the students utilize this equipment.

Prof. WHITAKER: It must be apparent that such a laboratory course as this could not be successfully given unless you had a considerable number of consecutive hours, and I propose, therefore, that the major portion of this work be given in the summer, so that I can get the boys in at 7 in the morning and keep them until 6 at night, if necessary for eight weeks.

Prof. SMITH: I am delighted to know that some institution has taken up the teaching of chemical engineering in this way. It has been my idea for years that it is the right way to teach the final steps in the engineering student's education. I have had some such course in metallurgy as this, at Case School, for a number of years, and I have found one important item of advantage already in it that Prof. Whitaker did not mention. I added a blast furnace. I know Columbia University does not believe in their metallurgy department having students operate a blast furnace, but I do not believe it bad to teach them the operation of it, and I do think they get in that operation a great deal of enthusiasm for their work. They think they are doing something, and they go back to their lectures with new ideas, which is very satisfactory to their instructor. The plan of giving students knowledge of these fundamental principles is most valuable; if they do not have this when they go into the work, the workmen, foremen and everybody connected with the place have a contempt for the college man, and he must stay there three or four years before he can live down that handicap that he gets the first few years of his employment in the factory. If he can give the idea that he knows either how to put together a lot of pipe, or lace a belt and put it on to a pulley, and a few fundamental things of that kind, the workmen there think that he knows something. They think if he cannot do that he is absolutely no good, and if he can do that he gets their permanent respect at once.

Secretary OLSEN: I would like to ask Mr. Whitaker if this work comes during the summer following the fifth year?

Prof. WHITAKER: Yes.

Secretary OLSEN: I would like to point out in this connection

that in this course, the student who undertakes this work has had one year more of college work than is ordinarily undertaken at present, when the student goes into a business. That is, having finished five years of college instruction, he will have had more instruction in chemistry, and other sciences, than he would have had after the ordinary four years' course. This chemical engineering laboratory has introduced the idea that instead of the student going into the factory and making his breaks there, he will get his experience under the supervision of the college professor in that eight weeks' summer time, and he is better fitted for doing it by his college instruction, and goes into a laboratory equipped for him to study the factory operations.

I want to make this remark in addition. It seems to me it is a splendid idea. I think it marks an advance in chemical engineering instruction which, it seems to me, is of the greatest importance. I notice that the student will have had two weeks of factory inspection at the end of his fourth year. I also notice that in his fifth year he remains in college after he has had his summer of factory inspection. After he has had his summer work in this chemical engineering laboratory, he is then to have a sixth year of college instruction, and his college professors are going to have an opportunity of showing the relation between this and the factory, to point out the important steps and the relation between practice and science. So it seems to me after he has finished that sixth year he ought to be able to go out and be far more successful than any graduates that we have at present.

Prof. JAMES: I think Dr. Whitaker is doing a great deal of good in this pioneer work. He is working right along the lines of some dreams I have had in teaching students the fundamental operations of chemical engineering. We expect to do that in our institution as soon as we get money enough. I believe that for one to understand these fundamental operations is to a certain extent to insure that he will make a success the first year he is out of school. In other words, he will make good right away. I think he has a great deal better chance for success. That is one of the things that will result from work, in this course of chemical engineering. Another thing is that it is an opening wedge for research work in chemical engineering, as such. When the institutions of the country that can afford it will have such equipment

as that we will have research work that is greatly needed, perhaps large scale work that no one firm undertakes. I believe that various institutions will follow Prof. Whitaker's example, and I propose to follow it in my own place; that is one of the good results from his work.

Prof. WITHROW: I want to congratulate Prof. Whitaker on the progress he has made in this direction, and also to emphasize it as my own firm belief that this is in the right direction. I also want to especially commend what most of us who have had experience in teaching industrial chemistry have learned, and that is the importance of letting students learn by error. I think we, ourselves, learn more in consequence of the mistakes we have made than in any other way. I think it is extremely important at this stage that the student should learn these things.

Prof. WHITE: I think it is perfectly true that "nothing succeeds like success," and I have not the slightest doubt but that under Prof. Whitaker's direction this laboratory at Columbia University will produce noteworthy results. I am glad to have had Prof. Olsen emphasize the fact that this was a five year course and in general this method is not applicable to a four year course. I desire to question, also, whether it is not possible to do some of the things Prof. Whitaker emphasizes, without actually resorting to laboratory work. It does not seem to me absolutely necessary that a man should have to wait until he tries to produce a solution of aluminium sulphate in water to realize that on the technical scale you have to consider what your curve solubility is. By the time you study on a technical scale the manufacture of aluminium chloride, etc., you ought to learn that the whole system is based upon the knowledge of solubility curves in the same way as other salts. There a man will undoubtedly remember, after he has made a fool of himself, that he ought to have known, and it is a good thing to have as many mistakes behind you as possible before you go out into the world, where mistakes are very expensive, but it does not seem to me that for a four year college course a laboratory of this sort is necessary. I admit, it is a fine thing where a person is studying the multiple effects to be able to get down and see the nicety of it, and the layout of it, but the fear that I have for a laboratory of this sort is that if it is introduced in our colleges with a four year course the student will become so absorbed with

the machine that he will forget the work. In other words, I am afraid that unless it is managed very carefully it would degenerate somewhat into a kindergarten work. As a graduate department, I think it is splendid. I wish we could have one here, but I have no ambition to duplicate that plan in our four year students' course.

Dr. ARTHUR C. LANGMUIR: I think Dr. Whitaker is not only working exactly in the right direction but I wish he had 5 to 10 millions at his disposal, because I should like to see a laboratory equipped with not only evaporators, showing their practical effects, and centrifugal machines, etc., but the very best and latest types of machines on the market. I should like to see such an institution be able to afford the luxury of a scrap pile and scrap things if they became the least antiquated. It seems to me such an institution could lead the chemical industries, and be far in advance of many lines of chemical industry, and the students they send out in a majority of the cases would be able to be of some advantage to their employers and undoubtedly be able to point out many ways in which they could better their equipment and economize. I think that we will live to see such an institution before many years. I think that Dr. Whitaker is to be congratulated on having made the start that he has.

In regard to teaching the principles of the subject it does not seem to be wrong or impracticable to teach a man, for instance, the principles of evaporation by means of quadruple effect and interchange of heat. I do not see why we cannot understand the principles to better advantage there than he can with fuel and a barrel full of water. Such equipment is not so good.

President BAEKELAND: Prof. Whitaker in his usual modest way forgot to point out the most important piece of apparatus which is necessary to make a success of the equipment he recommends. We can easily purchase triple effect vacuum plants and any other machinery; any such equipment is not very expensive and will cost only ten, twenty or thirty thousand dollars. But the most important, the most indispensable part of the whole equipment is the teacher himself, and he cannot be obtained as readily as any piece of machinery, however expensive the latter may be. But it so happens that by sheer good luck, Columbia University has acquired a man who has earned his spurs in the industry, after

having been a teacher, and then was willing to drop his well paid position in an industrial enterprise, and work for a small salary for the good of his University, and of chemical education. This kind of "apparatus," for a chemical engineering course, a first class technologist, who is at the same time a good teacher, I fear very few universities will be able to purchase, as long as the inducements which are offered nowadays by our educational institutions are so small. I refer less to the slender salaries, which are paid to professors, than to the irksome feeling of a man of individualistic tendencies of submitting himself slavishly to the dictates of some Board of Trustees, made up of men whose mental qualifications are frequently subordinated to the fact that they are giving financial support to the institution. Such conditions frequently are more discouraging than a small salary.

I believe that an extended course like the one outlined by Prof. Whitaker, may, in the end, prove a gain of time to the student. Anybody who employs a young chemist knows what a job he has to get him through the first year of his employment and to turn him to some use. The young chemical engineer who knows it all, seldom earns his first year's salary, however small, although he frequently earns the contempt, if not the hostility, of every workman in the factory. The initial salary you pay him serves merely to find out whether there is some hope that, ultimately, he may be trained to amount to something. I would call it a "prospecting" investment. An extra year for a man taking Professor Whitaker's course would therefore seem a good investment for the student in chemistry. In his curriculum he will have encountered those elementary practical problems which occur in almost every chemical industry, and he will be better prepared to cope with them in practical life. The standpoint of Professor Whitaker seems too self-evident to need any defense whatever. Long ago, it was adopted by all other engineering professions. Why should it be different for chemical engineering?

I would like to point out to Prof. Whitaker that as far as my personal experience goes, some of the lectures which have done me most good in my connection with the business side of chemistry, were some lectures on political economy, which I followed when I was a beginning student. I think political economy is almost indispensable in a curriculum of chemical engineering, but probably Prof.

Whitaker has good reasons for not mentioning it. I desire however, to formulate a general criticism against any curriculum which extends over too many years; it kills the initiative and the spontaneous judgment of the better class of students; only the more passive or less alive students have the patience to stand long years of preparation before jumping into the harness of practical life, and personal responsibilities, where they can better sharpen their wits and test their abilities than by mere book-wisdom ladled out in school after cut and dried pedagogic rules. School education ought simply to provide the means and the enthusiasm for further intense self-education.

Prof. WHITAKER: I believe one of the greatest difficulties in getting the support for these chemical engineering laboratories is the chemists themselves. It seems to be so utterly impossible for them to agree upon what should and what should not be done. One is afraid to ask the support of people who have means, for fear they will consult some other chemist, where the chances are about even that the other chemist will tell the capitalist that you are crazy. I cannot help but think that whatever we do we might agree and concert our actions on this problem. As far as the cost of this work goes, I have figured a number of times, and think I have stated that one-tenth of one per cent of the net profits of the chemical industries of this country for one year would build and equip one of these laboratories, and that that amounts to more money than is now invested in the chemical engineering laboratories of America and Europe put together. Such a situation as that does not exist in any other engineering subject. I would submit that when you hire a mechanical engineer you can get him in the boiler house without blindfolding him, but when you hire a chemical engineer many have not seen a filter press and do not know where the slimes go in and where the filtrate comes out. If this scheme of education has come to stay and is filling the bill *for these other* engineering subjects, how can we avoid the final issue in chemical engineering?

Prof. WITHROW: Each one who teaches Industrial Chemistry or Chemical Engineering is naturally guided by his own practical experience in emphasizing or molding his course. For the last six years it has been impossible for me to secure in my own work at Ohio State University any adequate apparatus. I have not been entirely

discouraged by this feature, however, because many years before ever starting to teach the subject I had formed certain notions as to the importance of the proper point of view toward manufacturing problems as a result of my personal observation in the works, and consequently in my work here these ideas are emphasized very strongly without the use of much apparatus. My point of view has been to emphasize especially the feature that the problems of the factory are to be solved by the research spirit, and I have therefore made my own course entirely industrial chemical research. This has given me an opportunity of incorporating a great many of the ideas which have been mentioned in this paper and in the discussion. However, I must say that I sincerely feel the lack of chemical engineering equipment and as soon as it is possible to secure the same, I intend to utilize it, for I believe it has much value. To Professor Whitaker, therefore, belongs the credit for having been the pioneer in securing and emphasizing the importance of such equipment, especially showing the way to the fact that equipment can be obtained if we go after it properly.

Upon examining the new course as proposed at Columbia University, I do not agree with a former speaker that this course of Professor Whitaker's is necessarily a graduate course, for, while it is true that it comes between the fifth and sixth years, it will also be noticed that it comes only at the end of or in conjunction with the fourth year of Chemistry because no chemistry at all is contemplated in this course during the first year. I would, therefore, like to ask Professor Whitaker a question—"If Columbia University had not gone on the six year basis, would he not have endeavored to find room for such work as he has described in or connected with the old four year course?"

Prof. WHITAKER: Such a laboratory has a double function, first as a basis for instructing undergraduates in the methods of the Engineering of Chemistry, and second as a laboratory where research in Chemical Engineering may be conducted. We would undoubtedly have established such a laboratory and used it for both purposes.

THE NEED OF STANDARD SPECIFICATIONS IN OILS FOR PAVING BLOCK IMPREGNATION

BY JOHN HAYES CAMPBELL

Read at the Detroit Meeting, December 6, 1912.

The increasing use of impregnated blocks for street paving consumes larger quantities of creosote oil each year. These oils are supplied under specifications which prescribe definite specific gravities, fractions on distillation and insoluble matter, but unfortunately the mode of stating these constitutents varies greatly.

It is with a view of bettering this condition, and to suggest a Committee, to adopt a standard specification, that the data in this paper has been gathered.

The Wood Preservers' Association and the American Railway Engineering Association have formulated a method of fractionating creosote oils for tie and timber impregnation, and if the Institute of Chemical Engineers do the same for the paving block oils, the creosote oil industry would be well covered.

The Railway Engineering Association Specifications are as follows:

The oil used shall be the best obtainable grade of coal tar creosote; that is, it shall be a pure product obtained from coal gas tar or coke oven tar, and shall be free from any tar, oil or residue obtained from petroleum or any other source, including coal gas tar or coke oven tar; it shall be completely liquid at 38° C., and shall be free from suspended matter; the specific gravity of the oil at 38° C., shall be at least 1.03, when distilled by the common method; that is, using an 8 oz. retort, asbestos covered, with standard thermometer bulb ½ inch above the surface of the oil—the creosote, calculated on the basis of the dry oil shall give no distillate below 200° C., not more than 5 per cent below 210° C., not more than 25 per cent below 235° C., and the residue above 355° C., if it exceeds 5 per cent in quantity, shall be soft. The oil shall not contain more than 3 per cent water.

In addition to this standard grade, two inferior grades can be used in cases where the higher grade oil cannot be procured. As the second and third grades differ only in specific gravity and fractionation, the wording of the specification being the same as quoted above, I will simply indicate them to save space.

No. 2 grade specific gravity at least 1.03 at 38° C., liquid at 38° C. Fractionation: below 210° C., not more than 8 per cent, below 235° C., not more than 35 per cent, if residue above 355° C. exceed 5 per cent, it must be soft. Not more than 3 per cent water.

No. 3 grade. Specific gravity at least 1.02 at 38° C., completely liquid at 38° C. Fractionation: below 210° C., not more than 10 per cent, below 235° C., not more than 40 per cent, and the residue above 355° C., if it exceed 5 per cent, must be soft. Not more than 3 per cent water. (*Railway Age-Gazette,* Mar. 12, 1912.)

A much quoted specification follows:

Specific gravity at 38° C., at least 1.10 completely liquid at 25° C., and show no deposit on cooling to 22° C., not more than 3 per cent insoluble by hot continuous extraction with benzol or chloroform. Fractionation: up to 150° C., nothing must come off; up to 170° C., 0 per cent to 5/10 per cent; up to 210° C. 2 per cent to 4 per cent; up to 235° C., 6 per cent to 16 per cent and up to 355° C., 40 per cent to 55 per cent. Thermometer to be corrected for emergent stem, not more than 2 per cent water will be permitted.

Another specification much used by City Engineers is as follows:

Specific gravity at least 1.03 and not over 1.09 at 38° C. Insoluble in benzol and chloroform not to exceed 5 per cent. Not more than 3 per cent water; if it contains this amount, allowance must be made in treating for water. Fractionation up to 150° C., not to exceed 2 per cent; between 150° C. and 170° C., not to exceed 15 per cent; between 170° C. and 235° C., not to exceed 30 per cent; between 235° C. and 300° C., not to exceed 35 per cent all as dry oil. Residue shall be soft and adhesive. Shall contain about 25 per cent crystallizable naphthalene and at least 15 per cent anthracene oils.

Thirty-eight degrees C., or 100° F., is by agreement the accepted temperature for delivery of creosote oil, a variation of temperature

above or below this point being corrected at 1 per cent volume for each 22½° F. or 12½° C. This would make a variation of 0.0008 specific gravity for each 1° above or below 38° C. Specific gravities should be so determined and reported.

Insoluble matter is probably best determined by the method of H. M. Newton (Report Sixth Annual Meeting of the Wood Preservers' Assn. 1910). Two grams of the oil are weighed into a small beaker. Twenty volumes of benzol are added and the mixture thoroughly stirred. Two S. and S. No. 589 Blue Ribbon filter papers and 1 double thickness S. and S. shell, 80 x 22 mm. in size and tarred on a balance against two other filter papers and one shell, the papers and shells having previously dried in a desiccator. The tare is returned to the desiccator. The two filters are folded and one placed within the other, and the diluted sample filtered through them. The beaker is washed with an amount of benzol sufficient to transfer all particles to the filter papers. When the papers have drained, they are rolled up and placed in a Soxhlet extractor, and extracted with the filtrate from the filtration of the sample, until the solvent runs off colorless. It is then dried in an oven at about 80 to 90° C., together with the tare, and when dry is cooled in a desiccator, then weighed. Some of the original sample is in the meantime, filtered through a double filter of the same quality as previously used, no weight being taken of the papers. Two grams of the filtrate are taken, and subjected to exactly the same procedure as outlined above. Weight No. 1 gives weight of suspended matter plus anything thrown out of the oil under examination by benzol. Weight No. 2 gives the weight of precipitate by benzol only. The per cent of matter precipitated by benzol as obtained from No. 2 must not be calculated on the weight of filtered oil taken, but on the amount of unfiltered oil which would furnish this amount of precipitate, by the following data and formula:

X = free carbon or insoluble.
W = weight of unfiltered oil taken.
W' = weight of free carbon plus precipitate.
E = weight of filtered oil taken.
E' = weight of precipitate.

$$X = \frac{E}{E - E'}\left(\frac{100W'}{W} - \frac{100E'}{E}\right).$$

We now have specifications which have given satisfactory results in service, and methods for assaying the oils furnished under them; and are prepared to go into conditions confronting engineers in different parts of the country, when they invite tenders on paving material.

The coast cities can obtain the imported oils, which would meet all the conditions of the Railway Engineering Association specifications. The Eastern West, Middle West and West, east of the Rockies, must depend upon American oils, or mixtures of American oils with refined or crude tar in various proportions, and here is where the great cause of friction between inspectors acting for municipalities and paving block contractors has its origin. In the Middle West little if any creosote oil free from added tar is used for paving block impregnation.

The majority of specifications have a clause in them, stating that a pure coal tar product shall be used for oil, the cut off points of fractionation are stated and the amount of residue if above a certain point must be soft. The inspector, if he is at all conscientious, will balk at accepting that oil which he is reasonably sure contains added coal tar, without submitting the matter to the Engineer for whom he is acting. The Engineer has his street torn up, traffic is interrupted and he writes or wires to accept that which is positively prohibited by the clause under which the inspector is working. This places the inspector in a bad position in future contests. In view of these facts, the following procedure would avoid these contests. Apply the First Railway Engineer's Assn. specifications to the imported and American lighter creosote oils. Then make two other grades, No. 2, to contain a mixture of 70 per cent American or imported creosote oil and 30 per cent refined coal or coke oven tar, No. 3 to contain 40 per cent of American or imported creosote oil and 60 per cent of refined coal or coke oven tar.

This procedure would permit the Engineer to make the choice, best suited to his local conditions, and not force him to buy a spade under the name of a long handled shovel.

Appended are some assays of these various oils.

It will be noted that the straight American oil is below the first specification, but it was submitted for these tests by a reput-

IMPORTED CREOSOTE OIL, AVERAGE OF FIVE ASSAYS

Specific gravity at 38° C.	1.056
Suspended or insoluble	0.27%
Up to 200° C.	0.91 per cent
" 210° C.	3.68 "
" 235° C.	23.89 "
" 355° C.	80.87 "
Residue above 355° C.	19.13 "
Water	1.94 "
Residue pasty	

AMERICAN CREOSOTE OIL. 70% Oil, 30% Tar

Spec. grav. at 38° C.	0.9901		1.03		
Insoluble	0.28		1.22		
Water	none	none	none	none	
Up to 200	12.04	11.16	6.21	6.51	
" 210	17.82	17.70	10.58	10.49	
" 235	46.35	47.99	29.27	29.76	
" 355	91.17	93.67	74.34	74.17	
Residue pasty	8.83	6.33	25.66	25.83	Pitchy

	60% Oil and 40% Tar			40% Oil and 60% Tar		
Specific gravity at 38° C.	1.055			1.094		
Insoluble	.....			1.59		
Water	none	none		none	none	
Up to 200° C.	5.53	5.97		3.62	3.28	
" 210° C.	9.02	9.55		5.78	5.42	
" 235° C.	25.34	25.62		16.61	17.56	
" 355° C.	68.11	68.66		54.77	55.54	
Residue	31.89	31.34	Pitchy	45.23	44.46	Pitchy

UNREFINED TAR

Water	2.38
Up to 200° C.	7.59
" 210° C.	9.04
" 235° C.	16.92
" 355° C.	39.62
Residue pitch	60.38
Specific gravity at 38° C.	1.150

able manufactory with full knowledge that the sample was to be used for this work and is probably a good, average American oil.

Specifications using the term, if the residue exceeds 5 per cent, it must be soft, should state the temperature at which this residue should be soft. It would probably be better to say, when kneaded between the teeth, the sample must be soft and plastic, not crumbly.

SECRETARY: The author calls for a resolution or some action of the Institute in connection with the paper.

Mr. Campbell is probably very much interested in this, and

we have several other members who are very much interested in this line of work, and who are fully capable of acting on a committee of this kind, if the Institute should see fit to adopt the resolution which is suggested, and a committee be appointed to act with these other associations in this matter.

PRESIDENT: I would like to hear the views of the members present on this subject.

Dr. ITTNER: I do not know as it would be wise to adopt the specifications, because it seems to me that the people to adopt specifications are the users of creosote, and those who furnish it. I think we might recommend that specifications be adopted, but I do not think it would be wise for us to adopt the specification, as Mr. Campbell gives them.

Mr. BOOTH: If it is in order, I move you that the chair appoint a committee to look this matter over thoroughly, without any question of whether the recommendation shall be adopted or not adopted.

Dr. LANGMUIR: Second the motion.

PRESIDENT: The subject is open for discussion. Before opening the discussion I would like to remark that this looks to me like a highly specialized subject, and I am not aware that we have many members here who are sufficiently prepared to pass an authoritative opinion on this question.

Mr. BOOTH: In my opinion, the matter should not be settled by a committee of our members that you might choose. It is not necessary to settle it here or now.

PRESIDENT: I must confess that I do not know who are the members who are capable of passing on the subject.

Mr. BOOTH: The secretary so indicated while he was reading his paper.

PRESIDENT: Who are the members who are especially so qualified?

MEMBER: We have Mr. Dow of New York City, and Mr. DeCew, and I think Dr. Olsen knows a third man.

Secretary OLSEN: Dow is a very active man in this line, and Mr. DeCew, and Dr. Sadtler, and Mr. Peckham.

Dr. LANGMUIR: We have sufficient talent in our own association, but nevertheless it does not seem to me that it is the thing we should take up, but it is a thing for other organizations to take up.

PRESIDENT: Unless you are entirely convinced that it would be very useful, this is one of those responsibilities that we might just as well try to avoid. If we are going to nominate a committee, that committee will have to report on the matter, and the matter will have to come up again, and the committee must decide, and more than one evening shall have to be taken up to no purpose. If we undertake the subject we must do it thoroughly or not at all. Is it worth the trouble and are we qualified?

Dr. ITTNER: I read that paper coming in on the train and I confess that I think it is entirely out of our province to adopt specifications for a purpose such as he mentions there. I think it is the province of the civil engineer. I think the chemical engineers, those of us who are in that line of work, would probably be best able to recommend such specifications, but I think that it is out of our province to adopt such specifications and would be a great mistake for us to go so far as to adopt them.

Mr. BOOTH: It is not my idea to carry this to a conclusion and adopt such a specification, because I am opposed to it, but we must show our members courtesy, and that is the only reason I made the motion, and if the chair so feels I will withdraw the motion. If there is a better way, let some other member indicate it. I am absolutely neutral in the matter.

PRESIDENT: So am I. I have no reasons for deciding one way or the other. I thought it my duty to point out to you the conditions. There ought to be a sufficient reason for incurring the trouble and responsibilities which are involved here.

Prof. WITHROW: I have a feeling, too. The idea has been expressed that we do not want to go into the business of making specifications, but I wonder about one other point; I was in the manufacture of creosote oil, and I know those engineers who are supposed to be the ones who make the specifications make very ridiculous and impossible specifications, so it occurs to me, that it might be possible for the manufacturers to suggest something, as to whether there is or has been a difficult situation. I know when this industry comes up we will be in touch with it. The specifications were absolutely worthless to the most important users in the country at that time.

PRESIDENT: I think a way of settling that question would be to submit these recommendations to the Bureau of Forestry, where we

have to deal with people who are entirely independent of commercial enterprises, while, if we attempt it here there will be only two or three members of this Institute and every one of them may be connected with business enterprises..

Mr. BOOTH: I am perfectly willing to withdraw the motion if we can show this man proper courtesy.

PRESIDENT: I think the fact that we have submitted this matter to discussion is sufficient courtesy.

THE PRESENCE OF OXYGEN IN PETROLEUMS AND ASPHALTS

By SAM. P. SADTLER, Ph.D.

Read at the Joint Meeting with the Eighth International Congress of Applied Chemistry, New York, September 6, 1912.

The problem discussed in this paper may be stated as follows: Can the presence of oxygen in petroleum and asphalts be established by a direct method of ultimate analysis?

To get the full import of this question a few words of introduction are needed, bearing upon the subject of what those interested in the chemistry of petroleum and asphalt know with regard to this matter of the presence of oxygen in substances of these two classes.

Hoefer (Das Erdoel und seine Verwandten, 2 Auf., Seiten 55 and 56) gives a list of 59 ultimate analyses of petroleums from all countries. It is true that more than half of these are the earlier analysis of St. Claire Deville and Boussingault in which only carbon and hydrogen were determined and the balance needed to make 100 was assumed to be oxygen, but in a large number of more recent analyses, both the sulphur and the nitrogen when present have been directly determined and the balance then ascribed to oxygen. Notably in Russian oils and Japanese oils, both analysed in recent years and noting the sulphur and nitrogen, has this presence of oxygen been recorded.

Rakusin (Die Untersuchung des Erdoels und seine Producte, 1906, p. 77) also quotes more recent analyses of Russian petroleums by Charitschoff and by Nastjukoff, who find from 0.4 to 2.5 per cent of oxygen and, what is of interest, note that the percentage of oxygen increases in the heavy petroleums and residues with the specific gravity.

But we are not obliged to base our belief on the presence of oxygen in petroleums on calculations made from ultimate analyses. The discovery of the petroleum acids by Hell and Medinger in Roumanian oils and phenols and of the naphthene-carboxylic acids by Markownikoff and Oglobin has given us an explanation of the presence of oxygen and justified the assumptions made from the ultimate analyses.

With the natural asphalts, the case is different from that of petroleums. Although earlier ultimate analyses of asphalts gave large percentages of oxygen, it was because the presence of sulphur in them had not been recognized and the oxygen was supposed with the carbon and hydrogen to make up the ash-free bitumen. However, Köhler (Chemie und Technologie der Natürlichen und Künstlichen Asphalte, 1904, p. 81) gives several analyses of natural asphalts by Day and Bryant and by Kayser in which a small percentage of oxygen is given as present alongside of a larger percentage of sulphur.

Both Clifford Richardson and Prof. S. F. Peckham, eminent American authorities on asphalt, have taken the position that not only is sulphur a distinctive element for natural asphalts, but equally that oxygen is to be considered as foreign to natural asphalts.

Besides the natural asphalts, we have also to note the artificial asphalts, obtained from petroleum, either by simple removal of the volatile portions or by some form of treatment with oxygen or sulphur at high temperatures. To the first class belong such products as "D grade asphalt," made from California asphaltic petroleum, (Clifford Richardson, The Modern Asphalt Pavement, 2d ed., 1908, p. 263) and "Baku Pitch" (Ibid, p. 271) and to the second class Ventura Flux, Byerlite and Sarco Asphalt. Of these last mentioned products Byerlite and Sarco Asphalt have been made from liquid petroleum residuums by the action of a current of air, either drawn through or forced through at temperatures ranging from 380° F. (193.3° C.) to 500° F. (287.7° C.)

The action of the heated air may have two different effects (see Höfer p. 85) according to temperature and rapidity or quantity of air passed through. The oxygen may cause splitting off of hydrogen in the form of water with condensation of the hydrocarbons affected, or oxygen may be fixed, forming products

of oxidation which remain, in either case resulting in thick semi-solid or solid products. Not only would it be very desirable from a scientific point of view to determine which of these reactions has taken place, or whether both have united in the formation of the solid asphalt-like products obtained, but the matter has been the subject of investigation in connection with patent litigation over rival processes.

Of course, direct determinations of carbon, hydrogen, sulphur and nitrogen may and do leave varying deficiencies to be charged up to oxygen, but it would be desirable to be able to confirm these calculations by a direct determination of the oxygen in the product. No such method has thus far come into common use. The method of Baumhauer, neither in its earlier form nor in its later form, using a weighed quantity of dry silver iodate and requiring first a current of hydrogen, then of nitrogen and finally of hydrogen again, has not been favorably commented on by those who have tried it. The method of Mitscherlich of burning with mercuric oxide is also intended to give the oxygen at the same time that the carbon and hydrogen are obtained, but this method does not seem to have worked satisfactorily in the hands of those who have referred to it and has not been adopted by chemists.

The process which I desire to present to those interested in this subject is very simple in theory, although its execution is not free from difficulties and requires time for its proper completion. It is primarily, the invention of Dr. Wm. M. Cross, City Chemist of Kansas City, Mo., with whose permission I have worked upon it with a view of making it applicable to this class of products, and to whose courtesy I am also indebted for the permission to give publicity to these results. It consists in a combustion carried on in a current of dried and purified hydrogen gas, the front of the combustion tube being filled with iron wool, which, brought up to a bright glow and thoroughly reduced by the hydrogen, then acts as contact-substance and brings about complete reaction between the hydrogen and the vapors given off from the decomposing petroleum or asphalt, whereby any oxygen present is taken up in the form of water vapor passing on to be absorbed ultimately in a weighed chloride of calcium tube. In making the determination, hydrogen is passed very slowly through strong sulphuric acid, cal-

cium chloride and over phosphorus pentoxide into the end of the combustion tube containing the boat with the weighed asphalt sample, beyond which is a sufficiently long layer of iron wool. The combustion tube at the farther end is connected with a good-sized U tube containing purified asbestos wool or preferably spun glass and this to a weighed chloride of calcium tube for absorbing water. When the combustion furnace is first lighted, only that part of the tube containing the iron wool is strongly heated, the part containing the asphalt being kept cool. Hydrogen is then passed very slowly through the apparatus until the chloride of calcium tube used for collecting water has come to constant weight and so remained for some time. The part of the tube containing the asphalt is then increased in temperature very gradually until ultimately the boat and its contents are heated to the maximum temperature attainable and so held for a time. If the large U tube containing the asbestos or glass-wool is kept cool, no condensable vapors pass beyond, and if the current of hydrogen be continued a sufficient length of time after the full heat has been applied, it will take all water through as vapor into the weighed chloride of calcium tube. No trouble need be anticipated from the small amount of sulphur contained in the asphalt or petroleum product, because the heated iron wool is capable of taking it up in whatever form it is liberated.

After beginning my trial of the process with ordinary combustion tubing, I was led by reason of the necessity of keeping the portion of the tube containing the boat with the weighed asphalt cool, while the portion containing the iron wool had to be heated to a bright red heat, to try a tube of fused silica and have found this to possess great advantages. With a tube of transparent fused silica, some 30 inches in length, which I obtained from the Silica Syndicate, Ltd., of London, Eng., the iron wool can be brought to the desired heat, while the end of the tube containing the boat can be kept perfectly cool by water trickling upon it. By this means the rubber stoppers with which the ends of the combustion tubes are fitted can also be kept cool so that no overheating can take place.

I have not yet completed my analytical work upon the material taken to try out the method and prefer to reserve a complete illustration of the applicability of the method to both petroleums

and asphaltic substances for a fuller paper. I will, however, give two oxygen determinations in a blown petroleum-residuum, or so-called artificial asphalt.

Determination of Oxygen.	I. Grams.	II. Grams.
Weight of material taken	1.0065	0.9767
Water absorbed in $CaCl_2$ tube	0.0440	0.0394
Corresponding weight of oxygen	0.0391	0.0350
Percentage of oxygen	3.88	3.58

DISCUSSION.

Prof. EDW. HART: "I have had some experience in the determination of oxygen in metallic iron by passing hydrogen over it at high temperatures and I have very serious doubts of the success of the method, which a careful reading of this paper shows has been proposed by Dr. Sadtler, but not yet fully worked out. The use of silica tubes with hydrogen is not an undertaking that one may view without some apprehension in view of the fact that silica does not hold hydrogen very well at high temperatures."

Dr. H. KIPPENBERG: "The determination of oxygen in iron cannot fairly be compared with the determination of oxygen in organic substances, inasmuch as in the former case the accessibility of hydrogen to the material is much more limited. Moreover, if Dr. Hart says that silica tubes do not hold hydrogen very well at high temperatures, this has no bearing in the case, since it is not hydrogen but water that is to be determined. However, it would be well if Dr. Sadtler could produce comparative figures, for instance indirect analyses, proving the method of his direct analysis to be fairly correct. It would also be well to give references in regard to the findings of chemists of the practicability of the Bauerhauer and Mitcherlich methods (page 4).

Dr. SADTLER: While I am not yet ready to publish complete analyses of any of these oxygen-containing asphalts because the matter is still withheld from publication on account of its being connected with patent testimony, I can quote with regard to this direct method the analysis of one other product in which I obtained

4.17 per cent of oxygen by difference. The chemist to whom I referred in my article as having first proposed the process, Dr. Cross obtained 4.14 per cent by difference, and the direct determination of oxygen by this new process gave 4.01 per cent. I have since obtained other results, but as before stated, the analyses cannot be published as yet.

THE CHEMICAL ENGINEER AND INDUSTRIAL EFFICIENCY

By WM. M. BOOTH.

Read at the Detroit Meeting, December 6, 1912.

The chemical engineer can take his legitimate place in industrial affairs only when he begins to concern himself with values and returns and can transpose the signs and symbols of the chemist to the dollars, cents and percentages of the business world. The profession must necessarily include men with diversified mental attributes, experiences and education. Natural inventors, builders, executives, analysts and economists are examples of the men who will choose this branch of engineering as a life work.

The first and highest type of endeavor concerns itself with the invention of new processes and their perfection, later establishing useful industries. Relatively few men have the ability, courage and means to embark in totally new enterprises, and the larger percentage of those who do so, fail, because these three important elements are not properly balanced.

Not less important, but demanding a different type of mind, perhaps more strongly analytical in its nature, is the large and ever increasing field open to those who are able to improve or effect economies in the processes of firms already established. Closely related to this is the examination of new enterprises that seem to have merit and which need scientific assistance and capital for development. No greater damage has ever been wrought on American investors by any class of men than by the "new chemical," "private," or "secret process" promoters who have used the subtleties of the science as a basis for fraud.

Irrespective of the particular division of endeavor undertaken, the chemical engineer must fortify himself against all classes of misrepresentation, and must concern himself with questions of

process, cost, market, location and actual capital needed in any new industrial enterprise. No considerable investment should ever be made without a complete report from conservative men, who are familiar with the industry involved.

While the basic operations of activity include agriculture, mining, transportation and manufacturing, the latter is chosen as best exhibiting the use of the chemical engineer along the lines previously pointed out.

Those who have made a careful study of the splendid papers of Dr. Monroe and Dr. McKenna to be found in the proceedings of this Institute, have been impressed with the fact that nearly all of the manufacturing of the United States is carried on east of the Mississippi River, and that more than one-third of this is confined to New York, New Jersey and Pennsylvania. The following census report shows the increase in capital and people employed from 1850 to and including 1909:

	Capital Invested in Manufacturing.	Employees.
1850	$ 533,245,000	957,059
1860	1,009,856,000	1,311,246
1870	1,694,567,000	2,053,996
1880	2,790,273,000	2,732,595
1890	6,525,051,000	4,251,535
1900	9,813,834,000	5,306,143
1910	18,428,270,000	6,615,046

It will be seen that the investment at the latter date amounts to eighteen billion dollars and the number of employees to over six millions. To maintain the almost perpendicular increase in our manufacturing activities is the duty of the commonwealth, for no more useful type of industrial activity can be found, especially when our products can be placed in foreign markets at a profit.

The observations covered by this paper are necessarily personal and have accumulated during 20 years, 10 of which have been devoted mainly to the questions now to be discussed. The observations cover 53 "going" plants, representing 37 industries, and some others that have never gone further than a prospectus distributed by an ignorant or a dishonest promoter. Obviously, in a paper of this kind, a general outline only can be given. The power plant was discussed in my first and subsequent papers before the Institute.

In looking back over the years covered, it would seem as though

the period has been one of unexpected crises,—rising wages, unsettled market conditions, and acute competition. Among the 53 concerns above mentioned, there have been 8 failures, 2 have withdrawn from business and 1, only, has burned. The remaining industries are owned as follows:

Private	11
Corporations	19
Trusts	12

The failures were attributed to the following causes:

Two to incorrect conceptions of the costs and profits of a a business.
Two to dishonest employees.
Two to incompetent supervision.
One to insufficient working capital.
One to manipulation of the stock in New York City in 1907.

The superintendents of 20 plants have been forced out or discharged for reasons such as inattention to duty,—incompetency,—lack of knowledge of the business,—trust management, the introduction of new methods,—inability to pay dividends on inflated stock issue,—dissension among officers of the company, or inability to handle help.

As I understand the matter, a general efficiency survey of any business must recognize the following conditions:

External: Policy of the government toward an industry.
Capital upon which dividends must be earned.
Location.
Cost of plant and equipment.
Internal: Unit cost of finished product, subdivided as follows:

Raw materials	Supplies	Fixed charges: Interest
Labor	Office expenses	Depreciation
Power	Repairs	Insurance
Lighting	Sales	Taxes
Heating	Advertising	
Cartage, freight and express	Charity	

Considering the capital invested, and the importance of the manufacturing business to all the people,—laborers, tradesmen and stockholders,—the Government is bound to respect, foster and protect these interests at all times. It would manifestly be suicidal to introduce any highly protected industry into the United States pending our tariff changes.

The passing of the pioneers who built and operated our first mills has thrown the responsibility of management upon the shoulders of many men totally unfitted by experience or temperament to carry on the business. To add to this misfortune of inheritance, all basic industries have expanded enormously, requiring more capital than one family or group of men could furnish. This led to the general adoption, between 1880 and 1900, of the corporate idea. While of the greatest value as an industry builder, no more pernicious influence has entered the manufacturing business,—this conception and use of an artificial individual without responsibility.

Abundance of money in the banks, the abuse of personal credit, and easy bankruptcy laws have made it possible to squander the people's money without scruple. In several instances in my experience, good operating managers have attempted to maintain their profits on an inflated and unwarranted stock issue, and have failed. Good men, improved machinery and low operating costs cannot off-set unnatural overhead expense. "Good will" has no place on the balance sheet of a well-regulated and solvent business. Any venture in the elaboration of raw materials ought, when brought to a paying basis, to be able to return to the stockholders a net profit of at least 10 per cent. Some old-established lines of business net from 20 to 60 per cent annually, and two good years have often paid for plant and equipment complete.

Considering now the matter of location; the manufacturers generally follows his market. As the growth of the population of the United States is westward, it has been found expedient to move whole industries from Massachusetts and New York to Michigan, Illinois and Missouri. Relocation of furniture and wagon plants has been quite general, because it has been found cheaper to ship the finished product to the markets than to bring the raw material East. Changes in market conditions have compelled the agricultural implement manufacturers to locate in the Middle

West. Formerly the manufacture of shoes was a Massachusetts industry; now, whole towns in New York and Missouri are devoted to this business.

No better illustration of economic change can be cited than the conditions at my own birthplace,—a valley south of Utica, N. Y. On a stream furnishing from 60 to 120 H.P. at each plant, 16 mills were in operation from 1873 to 1885. These employed about 2000 people. Forks and hoes were made in 2 plants, wagons in 1, woolen cloth in 2, cotton cloth in 5, knit goods in 1, silk in 1, paper in 2, and sewing machines and mowing machines in 1. The movement of freight and finished products kept the railroad and scores of teams busy. Furnishing provisions for the employees in the mills afforded a living for many tradesmen.

One agricultural implement plant was burned; one was bought by a trust and closed; the paper mills have been idle for years; three cotton mills have gone out of business; one woolen mill was burned and the other was closed; the knit goods plant and the machinery plant burned; and the silk industry was moved to Philadelphia. To-day, there are only three really good companies operating in the valley. Originally humming with the whirr of spindles and clicking with the throw of the shuttles in the looms, the valley is now quiet, and agriculture is the main business. Those of us who lived there did not know why so many fires took our industries away. It was observed that no factories were rebuilt. I now know that it no longer paid to make woolen and cotton cloth, to spin silk, and manufacture paper in that locality. The peculiar elements which we term *economic conditions* took our prosperity away, with incalculable loss to all concerned,—stockholders, employees and tradesmen alike. The moss-grown walls to be seen on many swiftly moving streams are monuments that mark industrial change.

Capital is sometimes invested in new projects, the aim of which is the use of raw materials that are supposed to be abundant, but that are later found to be insufficient in quality or quantity. Cement plants along the Erie Canal in New York State represent this class and also illustrate the rapid changes that may enter the elaboration of a product. In this instance the use of shale rock has taken the place of marl and clay in the manufacture of cement.

Beet sugar factories were started at Lyons, Binghamton, and at Rome, N. Y. The cost of the plant in each instance was very large, people cheerfully investing their money, expecting unusual returns to agriculture. After several years of failure and loss, the entire project has been given up; the empty buildings now remain after an expenditure of not less than $2,000,000. Colorado and California produce beet sugar at a profit. The location of the industry in New York State was a mistake, but the stockholders had to learn this. In the meantime, canning factories have sprung up throughout central and western New York and the well-managed ones are in a flourishing condition. Cheap raw materials, plenty of help, excellent transportation facilities and a ready market are contributory causes to its success.

No science or art can determine with accuracy whether economic conditions are correct for the location of an industry. Accident or good business judgment may accomplish what statisticians and scientists cannot.

Cost of Plant and Equipment. With plenty of money at his disposal, the optimistic manufacturer is apt to spend too great a portion on buildings and equipment. Ample working capital should always be held in reserve. The amount thus employed will necessarily vary with the business, but from 25 to 50 per cent is commonly set aside for this purpose in smaller industries, the capitalization of which is from $50,000 to $200,000.

It is much better to build a plant in a modest way, anticipating growth, than it is to find capital for running a concern in the midst of business expansion; especially so, if stockholders have had to wait from 3 to 5 years without dividends.

Again, a very expensive plant may be erected with consequent large overhead expense, where the income does not warrant the outlay. I found it impossible to recommend the erection of a water-gas plant in a town of 3500 people. A canvass of all prospective users showed that not more than 2 or 3 per cent income could be expected. A similar plant in a town of about this number of people can positively pay no more than 2 per cent on the investment after all avenues for gas consumption have been thoroughly exploited.

Location. To aid those who wish to study the problems concerning location, I have made a list of the elements that seem important. These are as follows:

Accessibility of raw materials
Market
Transportation
Labor
Power
Water
Supplies
Climate
Hygienic conditions
Taxes
Insurance
Banking facilities
Heating
Lighting

Obviously, all of these details cannot be discussed in a general paper. The importance of one item, water, is taken up in the second part of this communication, to show how carefully capital should investigate a new location, when the growth of a business warrants a change or when misfortune requires it.

With a favorable location, a demand for the product, an honest stock or bond issue, a modest initial outlay for buildings and equipment, and sufficient capital for doing business, an executive should earn a fair dividend on the investment.

Internal Unit Cost. The ideal is the basis of our best effort everywhere. In manufacturing, this ideal is approached when the largest quantity of good goods is turned out at the lowest possible cost. To determine how to attain the above conditions is the duty of every executive. But the task is not a simple one, for book keepers are not statisticians and although all of the data necessary in connection with the proportioning of expense in the manufacture of an article may be derived, it takes a large amount of study to draw correct conclusions. No simpler expedient in my estimation has been devised in this connection than *unit cost.* What does it cost to produce a pound, a horse-power, a barrel, a yard, or a machine,—any single article, many of which are made each month or year? These items may be apportioned as percentages. The separate items under fixed and operating expense become factors of the total. As an example, we will say that the production of a machine has entailed an outlay of $25.00 and that this is divided as follows:

Raw materials	20%
Labor	40
Power	7
Lighting and heating	1
Transportation	3
Office expenses	2.5
Repairs	1.3

Sales	10
Advertising	5
Charity	0.2
Interest, depreciation, taxes, and insurance	10

This method shows at a glance that labor is a large item in the cost of this article and that any economy that can be effected in this department will make an appreciable saving in the total outlay.

Through the kindness of Mr. E. Durand of the Bureau of Census, I am able to present a comprehensive table illustrating the method above outlined:

This is a valuable guide in any efficiency study of production costs, although too many items are grouped under expense, which may be used by any executive to conceal exorbitant bills of any nature.

The actual cost of a case of tinned goods of the season of 1912 is divided as follows:

Raw materials	30.00%
Labor	14.40
Fuel	0.17
Freight and express	0.4
Maintenance	4.80
Sales	3.90
Advertising	0.90
Interest	1.90
Depreciation	1.80
Taxes and insurance	1.60
Boxes and labels	7.30
Loss on seed	5.80
Discount and brokerage	3.10
Expense	8.70

The remaining items are not considered useful in this paper. The tin container itself includes a cost of 85 per cent for materials and 15 per cent for labor.

A woolen mill owner and operator furnishes the following table:

Materials:	Raw stock	Labor:	Office	Expense:	Insurance
	Soap		Factory		Taxes
	Dyes		Overseers		Charity
	Wool				Executive salaries
	Coal				Depreciation
	Wool oil				Repairs
	Cotton and shoddy				

STATISTICS OF MANUFACTURE. (Thirteenth Census, 1910, page 30)

Industry.	Per Cent of Total Expenses Reported.			
	Salaries.	Wages.	Materials.	Miscellaneous Expenses.
All industries	5.1	18.6	65.8	10.5
Agricultural implements	8.6	24.3	51.1	16.0
Automobiles, including bodies and parts	4.5	23.1	62.5	9.9
Boots, shoes, including cut stock and findings	3.9	20.6	69.6	5.9
Brass and bronze products	4.1	17.3	72.6	6.0
Bread and other bakery products	4.0	17.4	69.9	8.6
Butter, cheese and condensed milk	1.4	4.3	91.0	3.3
Canning and preserving	5.6	13.5	72.0	9.0
Carriages, wagons, and materials	5.7	27.0	58.9	8.4
Cars, general shop construction and repairs by steam railroad companies	4.3	44.7	49.2	1.8
Cars, steam railroad, not including operations of railroad companies	4.3	23.0	66.7	6.0
Chemicals	6.5	15.0	68.2	10.3
Clothing, men's, including shirts	5.2	20.7	57.9	16.2
Clothing, women's	6.0	23.0	61.1	9.9
Confectionery	7.6	13.1	67.9	11.4
Copper, tin, and sheet-iron products	5.8	22.4	63.7	8.1
Cotton goods, including cotton small wares	2.6	24.0	66.9	6.5
Electric mach., apparatus, and supplies	10.0	24.5	53.8	11.7
Flour mill and grist mill products	1.5	2.6	92.8	3.1
Foundry and machine shop products	8.7	29.8	50.1	11.4
Furniture and refrigerators	7.3	30.8	51.0	10.9
Gas, illuminating, and heating	10.9	18.4	46.2	24.5
Hosiery and knit goods	4.4	25.5	62.7	7.4
Iron and steel, blast furnaces	1.8	6.8	88.4	3.0
Iron and steel, steel works, and rolling mills	2.9	18.3	73.9	4.8
Leather goods	7.2	19.3	64.6	8.9
Leather, tanned, curried, and finished	2.2	10.5	81.2	6.1
Liquors, distilled	1.0	1.6	18.4	79.0
Liquors, malt	7.6	13.7	32.2	46.5
Lumber and timber products	4.8	32.0	51.0	12.2
Marble and stone work	6.7	44.8	39.4	9.1
Oil, cottonseed and cake	3.1	4.3	87.7	4.9
Paint and varnish	9.3	7.4	71.1	12.2
Paper and wood pulp	4.0	17.2	69.7	9.1
Patent medicines, compounds, and druggists' preparations	14.9	8.7	44.1	32.4
Petroleum, refining	1.8	4.4	89.6	4.2
Printing and publishing	16.7	26.6	32.6	24.1
Silk and silk goods	4.2	21.8	60.8	13.2
Slaughtering and meat packing	1.5	3.9	91.3	3.3
Smelting and refining, copper	0.7	3.8	94.4	1.1
Smelting and refining, lead	0.9	3.4	94.8	0.9
Sugar and molasses, not including beet-sugar	0.9	2.8	92.6	3.7
Tobacco manufactures	4.6	19.0	48.4	28.0
Woolen, worsted and felt goods and wool hats	2.6	18.7	72.9	5.8
All other industries	6.4	21.1	62.1	10.5

Percentages were not given but the total annual expense including the three subdivisions above shown, is divided by the number of yards of cloth turned out to determine the cost price per yard.

I am familiar with the actual cost of reducing a ton of garbage by the naphtha process. This, on a percentage basis is as follows:

Labor	47.43%
Coal	20.81
Pressing	12.37
Filter cloth	2.89
Gasoline, oil, and light	8.00
Freight	1.77
Superintendent	3.97
Taxes	0.45
Interest	0.28
Office	0.24
Commissions and analyses	0.74
	99.94%

Labor is shown to cost an excessive amount. The gasoline item needs investigation.

The following elements compose the cost of a small copper instrument:

Labor	61.62%
Raw materials	27.41
Burner	6.03
Hanger	1.31
Polishing	1.64
Support	0.54
Paint	0.22
Screws	0.22
Lacquer	0.55
Bolts	0.33
Solder	0.11

Obviously, the labor cost of the instrument is excessive.

The official having charge of the cost department should ascertain at fixed intervals, by inventory and from records, the exact unit cost of any or all articles produced. This information should be made a part of a blue-print chart, carrying at the left a list of the items composing the record and a continuous line, showing the fluctuations in the cost of each, from month to month. If any one of these lines rises from causes beyond the control of the management, others must fall, if the price of the finished article

is to be maintained. Heating and lighting will fluctuate with seasonal changes; interest and depreciation, charity and office will remain practically horizontal. Unless the industry controls its own raw materials these will usually increase in price; labor of all kinds increases; power is more expensive because coal is higher in price and water power has in many plants been replaced by steam. There is a very gradual rise in transportation costs and in supplies; taxes and insurance are constantly increasing everywhere.

With such records available, the operating manager is able to determine where efficiency methods could be applied to advantage.

In a former paper before the Institute, I have discussed "Power" from a percentage basis. Since that time, the use of electricity has become quite general and the H.P. year cost has been considerably lowered in a large number of plants. The remainder of this paper will be devoted to the purchasing and storing of supplies.

Stock and Supplies. The raw materials needed in operations of the manufacturer are usually closely bought and economically handled. I have found this particularly true in woolen, cotton and paper mills.

Every successful enterprise requiring raw stock must include an expert buyer of large experience, whose business it is to make a study of market conditions and fluctuations. Ten years ago, much more practical experience was required to fill such a position than at present. This has been brought about by the general introduction and use of specifications. One by one, natural products have been standardized; coal, wool, cotton, ores, oils, paper stock, iron and steel and natural earths; these are a few of the hundreds of raw materials used by manufacturers. Physical and chemical standards have been set to replace the guess and estimation methods that came from experience and that are often wide of the truth. A relatively inexpensive man, who has a testing laboratory at his disposal can determine the actual value of raw stock much more accurately than some high priced man can guess at it. Two items on our cost sheet can be turned downward and kept there at relatively small expense.

Unless the specification idea is carried throughout the mill to include the finished product, the work is incomplete, for the adoption of such a system invariably improves the quality of the goods turned out. For example, every piece of wood, of composition and

metal that goes into an automobile or locomotive should be of the best quality, proved to be so by actual experiment. This rule holds for manufactured products generally.

One of the weakest points in the personnel of the mill organization to-day is the purchasing department. I refer more particularly to those in charge of the purchase of the supplies. This important branch of the business is often left to incompetent clerks, who antagonize salesmen generally and who buy from men whom they get the greatest possible return,—gifts, dinners, an occasional trip, or even money. Honest traveling men avoid such purchasing agents, to the permanent loss of the business.

A shrewd salesman may spend from six weeks to six months in placing an order for expensive equipment that a concern never needed and should not buy. In my experience, men totally remote from the ordinary purchase of supplies should be employed in connection with new and valuable equipment. Consulting engineers can act to good advantage, turning in reports that show the general market conditions, kinds of apparatus or material available, with the experience obtained from the use of these in other plants.

Supplies. Every manufacturer uses mixtures and compounds the exact nature of which he has no knowledge—oils, dyes, fillers, adhesives, cleaning agents, waxes and polishes, powders and salts. In some instances, such materials have a total cost of $2000 per month. Many simple substances are sold in large quantities, at inflated prices. For one concern, I was able to lower the cost of a special substance brought for $50 a ton, by substituting the same material from another source at $15 per ton. Good business requires a knowledge of supplies and their component parts, for the purposes of keeping the cost down, for the protection of workmen, and to guard against fire.

No feature of factory economy should be as closely watched as the storeroom. This should be separate, light, orderly and so arranged that many articles varying in size and shape can be found quickly. A store keeper should be in charge every moment of the working day, and should be held responsible for all stock handled, distributing this over a counter only, never allowing workmen to come behind this. All orders should be signed by proper authority and a carbon copy of each transaction kept. Tools, in particular, and all stock that can be used about home, barn or

garden will mysteriously leave the plant, a few cents worth at a time, if a way is found to handle the matter with an easy conscience.

Summary. In the foregoing paper, I have attempted to point out to you certain methods that can be adopted in the conduct of any manufacturing business. The days of large profits, cheap raw materials and labor have gone for good. With increasing prices and competition, all executives must be economists as well. Efficiency, in its broadest sense should include promotion, capitalization, location, organization, equipment and operation. Great stress has been laid on the efficiency of labor to the exclusion of matters of equal or greater moment. An exact knowledge of unit costs will reveal the weak points in any plant. The so-called efficiency engineer, who spends three or four days in a concern and antagonizes everyone from the bosses to the office boy, can accomplish little good. Weeks and months of study are required to get at the details of the business; to make improvements is a still greater task. The good-will of the employees must be gained and kept to make any progress in economy studies. Discussion and argument naturally follow any change in policy. The man who makes the change should be on the ground to defend himself and drive home the facts as he sees them.

WATER FOR INDUSTRIAL PURPOSES

By WM. M. BOOTH

Read at the Detroit Meeting, December 6, 1912.

Water Supply for the Manufacturer. Before locating a new plant or industry, a large amount of preliminary information is necessary. Having decided that market conditions, transportation, labor and power are available and acceptable, a thorough study of water conditions must be made. This should determine the quantity, quality, and cost of this material necessary in the production of power, for strictly manufacturing operations, for drinking, for cleaning and for fire extinguishing purposes. Sum up all possible requirements and add from 50 to 100 per cent for emergencies and for growth.

If possible, two independent sources of supply should be obtainable.

Information of the character demanded can be obtained from a commercial laboratory that has specialized in this direction, or from a consulting engineer who has a laboratory at his disposal. Such a study should be begun at least a year in advance of building operations. Seasonal changes are such that both quality and quantity of water may vary greatly from month to month, if the proposed source is a river, small stream or spring.

Having employed a man or company to make the necessary observations and analyses, the proposed sites can be visited and the requirements pointed out and discussed. If water is to furnish the power of the mill and maximum and minimum flow data are not available, they must be obtained from the government or from original experiments conducted with a weir. Special forms of this device are now sold with recording gauges that read in cubic feet per second. If records have not been kept and the expense of the instrument is more than is thought necessary, actual velocity and

depth studies can be undertaken at stated dates each month. If the minimum of these readings shows the passage of the required volume of water, the questions of quantity are dismissed for power, fire and cleaning.

Such water is rarely exactly fitted for power and technical purposes. There is, however, a considerable area of granite or related rock in the United States, the run-off from which is soft and clean. The Adirondacks, the White Mountains, and the Catskills all afford an abundance of soft water. The plants along the streams running from these mountains have many advantages due to this kind of water supply. I have found the hardness to average about fifty parts per million.

Shale rock areas in New York State at an altitude approximating 1400 feet also afford very good water, with a hardness between 100 and 120 parts per million. Limestone areas generally produce *hard* water. Such a belt runs through the United States from eastern New York to the Mississippi River and south, hardness 150–2000 parts per million. Before use in a power house, this class of water should be softened by chemical means. The cost of treatment varies from the fraction of 1 cent to 20 cents per thousand gallons.

The quality requirements for manufacturing purposes vary with the nature of the business. Textile mills demand a great volume of soft water free from iron and sediment. Canned goods concerns need a water under a hardness of 170 parts per million, clean and sanitary, low in organic nitrogen. Power producers and steel plants need large volumes of soft water, free from sediment and oil, and cold for condensing purposes.

Many industries use water for rinsing. Laundries must avoid water containing iron; in addition this should be soft and clean. Nor can iron tanks be used in storing water for laundries; but wood and concrete are always available.

In fact, very few operations of manufacturers require hard water, and in such cases the necessities are generally well understood.

If a stream cannot be appropriated for water supply, a lake may be considered. The Great Lakes serve hundreds of plants. The water has a hardness of from 90 to 120 parts per million. As a cheap source of water of enormous volume they are unexcelled.

All lake water carries sediment which can be treated according to the needs of the business. One grain of alum per gallon cleans the usual run of lake water at a cost of less than $2.00 per million gallons.

Smaller lakes in the Eastern States are usually on high ground and necessitate a water works system between the source of supply and the plant on the railroad. New York State includes within its borders hundreds of such natural reservoirs. The water has a hardness, approximately, of 100 parts per million.

Next to lakes, small streams and isolated springs may be considered. The annual maximum and minimum supply must be definitely determined before any plans are made to use such water. Large streams with a large average flow fifty years ago are often dry in midsummer now.

Perhaps the industry does not need a large volume of water daily, or must be situated in a town or city. When there is no choice of location, bad water must be corrected mechanically or chemically. If there is a choice between two or more towns, the question from the water investigator's standpoint is what kind of a supply and what equipment exists at each place.

A complete history of the water works company, plant and equipment should be worked up,—short, but showing location and extent of watershed, storage capacity, piping, pumping plant and the financial standing of the company itself. The quality from a sanitary, mineral and bacteriological standpoint should be made a part of the record,—the result of personal investigation and not from published records.

No modern mill superintendant can tolerate a scant supply or a poor distribution system. We all know of towns where the water pressure varies from 20 pounds to zero. Insurance rates are unduly high and fire losses are numerous. Few small towns can supply water in quantity sufficient for big business. My clients operating canning factories require from 50,000 to 100,000 gallons of good water per day for special uses; woolen and cotton mills from 1,000,000 to 5,000,000 gallons per day; paper mills from 2,000,000 to 5,000,000. Canals and rivers flowing through towns are the only inducements for large users. City water costs from 3 to 15 cents per 1000 gallons; 10 cents is not unusual. The bill for this item alone may amount to $30 to $50 per day. Such a fixed factory

cost is not unheard of but it cuts down profits with regular and insistent demands.

If a town will furnish clean, soft water at 10 cents per thousand gallons to a concern using not more than 50,000 gallons a day, the chances are that the use of such is desirable rather than an attempt to find other sources with certain overhead expense and uncertain results.

This statement applies also to wells, fully discussed in my previous paper before the Institute.

Drinking Water. No two sanitarians will agree concerning the standard to be set for potable water. In this paper I suggest three types of water with possible limits of purity. Many thousands of people are drinking each of the three grades daily, with apparently no ill effects.

These tentative standards are as follows:

	Parts per Million.		
	I.	II.	III.
Free ammonia	0.02	0.05	0.10
Alb. ammonia	0.08	0.10	0.15
Nitrates	1.00	2.00	2.50
Nitrites	0.000	0.005	0.01
Chlorine	1.00	10.0	20.00
Oxygen consumed	1.00	2.00	3–5
Total solids	100.00	500.00	500–4000
Colonies per c.c.	100.00	500.00	1000–20000
B. coli	0.00	Presence in 10 c.c.	Presence in $\frac{1}{10}$ c.c.
Sediment	none	slight	heavy
Color	none	slight	yes
Odor	none	yes	yes
Temperature	winter above 45° F.	40° F.	very cold
	summer below 60° F.	80° F.	very hot

With increased attention to sanitary details everywhere, the manufacturer can well afford to add a clean, pure supply to his factory equipment. If it is impossible to obtain potable water, it should be prepared. In case the supply is muddy, it should be filtered; if polluted it should be passed through sand and charcoal or distilled and then passed through charcoal. It must be remembered that water distilled from an impre source, organically, carries ammonia and sometimes other gases. Charcoal gives such

water a pleasant taste and removes odor. Do not attempt to condense steam from an ordinary boiler for drinking purposes. Rather pass steam through a copper coil in a tin lined kettle. Condense also in tin. Such water must be cooled before drinking.

When a manufacturing corporation can afford to do so, it should build and operate its own water works. Ten, fifteen, or twenty miles is not a prohibitive distance to go for a good supply. The initial expense will be small compared with the additional resources of the plant that has all of the clean, soft water needed.

To accomplish such a purpose, it may be necessary to buy several hundred acres of cheap land. This should be fenced and all people and animals kept out. Having an abundant supply, a corporation may add to its income by selling water.

If water of a poor quality, but soft, is available near the concern, a purification plant on a large scale can be built to good advantage. Here water may be filtered or softened to the degree required by the average use to which it is put.

DISCUSSION.

PRESIDENT: Gentlemen, Mr. Booth has presented to us a lot of simple facts which although self-evident to many of us, are unfortunately very often overlooked, especially by chemists. I personally remember very well an incident where electrical power was offered by two localities. In one it was offered at a price of 50 per cent below that of the other, and the amount of power under consideration was about ten thousand horsepower, which meant a difference of many thousand dollars a year for the cost of power, and yet it took fully two months of calculation and investigation in order to find that the power which would initially cost so many thousand dollars a year more, was finally the cheaper, because unavoidable interruptions of the cheaper power swallowed up the general efficiency, so as to offset any initial benefit of cheaper power rates. There is one subject, which Mr. Booth has not mentioned, and which in some instances has caused considerable trouble, namely, the transplantation in this country of foreign industries raised and developed in Europe. For instance, industries which have been successfully carried on in England, France and Germany and which were tried in this country with practically no doubt as to their success, because

the promoters believed the conditions here would be the same, and where the enterprises did not succeed because the originators did not take into consideration the extreme changes in the climatic conditions of our seasons. In winter time we have an unusually dry climate, incomparably drier than in England or the European continent. Then again, in summer time, some industries are impossible here on account of abnormally high dewpoint or exaggerated amounts of humidity in the air. Around New York in July and August, and even during the first days of September, there are times when the dewpoint goes as high as 70° to 74° F. Such extreme conditions of temperature and dewpoint are practically unknown in Europe.

We still have a few minutes to discuss this subject. Is there anybody who desires to make some remarks.

Prof. BARTOW: I would like to say a word in regard to the second part of the paper. I think the suggestions in regard to the need of good water supply is important. I have recently had my attention called to a factory located in a town where the city authorities agreed to furnish them all the water they wanted, and did not take into consideration the fact that this one factory would need more water than the whole city supply, and the fact that the city was itself quite short of water. Also the lack at times of appreciation on the part of municipal authorities and others is sometimes brought out when, for example, the mayor of a city states that he does not believe that a good water supply can be furnished through city mains, and when he goes to cities with different water from his own town (he, by the way, does not have good water in his own town), he cannot drink, and says that he would never in any town drink the water which was furnished, no matter how good it might be.

Mr. BOOTH: There is a business which is on very good footing in Sweden, and a man of my acquaintance went abroad and brought back samples, and interested 30 men, and I was one of the unfortunate 30 men who invested some of my money to help start that industry in this country, capitalizing for a hundred thousand dollars, $63,000 paid in, and we put up a building and had $18,000, to put in the business. We had no market in this country, and endeavored to start a market. The $18,000 disappeared, and then they came to me and said, "Your stock is no good, and we will pay 30 cents on the dollar for it." The concern failed, and I was glad

to get that out of it. It only goes to show what the dangers in manufacturing are. The point in my paper is that manufacturing is an enormous and growing business. It has a future in this country. The efficiency of the mines is going down, as far as the amount of gold that we can get out of the earth is concerned. That line is going down, and one of the greatest industries, manufacturing and inventions, must replace what we have lost in the natural resources, and so I am bringing to you chemists and chemical engineers something which you should be interested in, as chemists, and as inventors.

There is one point which deserves careful consideration by all having experience in business matters; that is, that the best business can be swallowed up by over-capitalization, and that very often happens in this country. There is no limit to the amount of water you can pump into any organization, and many people, especially chemists, do not always realize that, however good a proposition may be, it can be simply annihilated by the unscrupulous promoter who pumps in water and wants to capitalize everything in it, and then leaves the whole thing high and dry, and then you must wait until bankruptcy sets in, and all the time you have become lukewarm on a very good enterprise, which for this reason becomes impossible. For instance, inventors having a patent or invention to sell, often do not realize that there is a limit to the value of an idea or invention. The best patent in the world is not worth more than a certain amount of money, and the most extravagant starts have been made in some industries, and that is mainly due to the fact that the supervision of the firms or incorporations in this country is very imperfect as compared with Germany. In Germany you cannot begin to capitalize everything—water, air, pipe dreams and rain drops. In some cases where firms pay a very fair sum of money for a patent, the patent is put down right away in the assets, "Value 1 mark," because if they did not do so the state would have said, "Well, you put that patent valuation 12,000,000 marks, now where is that value?" That would have deducted from the profits. It was better to start with a valuation of 1 mark. When people begin to speak of good will and the supposed value of a patent, there is no limit to the imagination of a promoter, which is generally very abundantly supplied, especially if the patent is the main commodity, with which to get the money of some people into his hands.

THE AVAILABILITY OF BLAST FURNACE SLAG AS A MATERIAL FOR BUILDING BRICK[1]

By ALBERT E. WHITE.
Of the University of Michigan.

Read at the Detroit Meeting, December 4, 1912.

There are three main products made in a blast furnace; one is pig iron, another is the so-called waste gas, and the third is slag. The first of these products meets a ready market and is the primary object of the smelting operation. The second of these products, the waste gases, have of late years been the recipients of a considerable amount of study looking to their complete utilization. Waste gases have been used for many years in heating hot blast stoves, which in turn preheat the air blast which enters the furnace. Only about one-third of the energy of the gas is used in this manner. The remaining two-thirds of the energy in the gas is already partly utilized and will probably be ultimately almost completely converted into power, through the agency of gas engines.

But how shall the slag be utilized? Various methods have been proposed for its efficient utilization, but none, at the present time, have received any great amount of approval. This, at least, applies to those methods which have aimed to utilize the great bulk of the slag.

The production of blast furnace slag in the United States at the present time is about 32,000,000 tons a year. The significance of this figure becomes more apparent when we consider that it is almost as great as the tonnage of steel produced.

The most important single utilization of the slag is as one of the raw materials entering into the composition of Portland cement. This industry cannot well utilize, however, the slag obtained at the time of casting because such slag carries small iron particles. Nor can it use to advantage slag high in sulphur, or of unsuitable

[1] Permission must be obtained from the author for making abstract or reproduction of this article.

composition. These conditions probably render it difficult to use over one-third of the slag in cement manufacture.

The annual production of cement in this country is about 75,000,000 barrels of 380 pounds each which amounts to 14,250,000 tons. Portland cement made with slag carries roughly 50 per cent of its weight in slag material so that one-half of the weight of cement made in this manner represents the weight of the slag which could be thus used, which is 7,125,000 tons.

According to the estimate given above one-third of all the slag or 10,000,000 tons might be used for this purpose. This means that the cement output of the United States might increase 50 per cent and all of it be made in part from slag while still using only one-third of our present slag production. Although the production of cement composed in part of slag is steadily increasing, it is not at all probable that it will ever displace clay and shale altogether as raw materials.

It was also suggested, at one time, that slag spaulls would make good railroad ballast, but, after 10 or 15 years of trial, the railroads have rejected this material because of the tendency of certain slags to fall to powder and cause a dust nuisance to the travelling public and, worse than this, numerous and serious track troubles.

Slag can readily be converted into mineral wool, but from the tonnage standpoint there is an annual demand for but a relatively small quantity of this material and thus one can easily appreciate from this same point of view how really insignificant is such a utilization.

When the problem was first given to the writer for investigation by a firm with whom this question is a live one, he was cautioned that the utilization he should propose must be one which would use a large tonnage. The one at first glance which presented the best possibilities was that of converting slag into a paving brick. Good paving brick is made from the slag of certain blast furnaces in England and on the Continent by casting the slag into blocks and annealing the product. Many attempts were made by the writer to convert the slag at his disposal into paving bricks but all attempts were failures. Some of the bricks broke up on cooling and those which remained sound were so brittle that they would usually break if dropped on the floor. The reason for this failure to make good paving bricks is not yet thoroughly understood. Mr.

E. C. E. Lord, Petrographer for the United States Government, has been working on the mineral constituents of slag for a number of years attempting to determine why the slag made in the blast furnaces of the United States cannot be made into a paving brick. So far as the writer is aware Mr. Lord has not yet been able to suggest a method of furnace operation feasible under American commercial conditions which will produce slag suitable for slag paving bricks.

It seemed more promising to attempt to use slag in combination with a small percentage of lime as a building brick, in a process similar to that employed in the manufacture of sand-lime brick. As at least 95 per cent of the constituents of the brick would be slag there was but little question but that, if successful, the process would afford an outlet for a large tonnage of slag.

The process in general for the production of slag-lime bricks should be nearly identical to that employed in the production of sand-lime bricks. Since this latter process is more or less well understood time will not be taken to go into details regarding it. One can readily comprehend that there is nothing essentially new about the idea. Because of the small quantity of slag-lime bricks made it was not possible to follow out in its entirety the standard sand-lime brick process. Since this was so, the method used in making these slag-lime bricks will be outlined.

The slag as it came molten from the furnace was dropped from the slag trough through jets of water into a granulating pit in accordance with usual practice. The finely granulated slag shovelled from this pit was dried and shipped to one of the most modern sand-lime brick plants in the country where the experimental slag-lime bricks were made. It was recognized that there would be no difficulty in getting combination between the lime and the slag, for slag is more reactive than sand. The question resolved itself into so correlating the grain size of the slag particles and the pressure employed in forming the brick as to get a finished brick of best physical properties. Eleven sets of brick were made. There were six sets in Lot 1, and in the main all six sets were of nearly identical composition and structure. There were two sets in Lot 2. These differed chiefly from Lot 1 bricks in the amount of pressure to which they were subjected for Lot 1 bricks were made under a considerably greater pressure than Lot 2 bricks. In

Lot 3 are found three sets of brick. This was the last lot of bricks made and the experience gained in the making of the first two lots proved of great value. For this reason the quality of the slag-lime bricks in this lot was superior to that of the bricks found in Lots 1 and 2. Because of these conditions the data relating to the composition and method of manufacture of Lot 3 bricks has been chosen rather than the data of either of the other two lots.

The lime and finely granulated slag were reduced to fine powder by grinding in a ball mill. A portion of the coarse granulated slag and, in addition, in one of the tests, some river sand, was then mixed with these ground products. A rough sizing of the constituents of the three sets of bricks found in Lot 3 gave the following results:

	I.	J.	K.
Lime, finely ground	7.3%	7.2%	10.0%
Slag, finely ground	34.4	34.4	35.0
" on 30 mesh	58.3	.....	50.0
" on 40 mesh	.....	58.4	
River sand	.....	.....	5.0

A small amount of water was then added to the mixture. The material was then fed into the brick machine and in this series in such volumes as to give the maximum pressure which the brick would stand without its showing air cracks when removed from the press. They were steamed for 10 hours at a pressure of 125 pounds per square inch.

Their analysis is as follows:

ANALYSIS OF LOT 3 BRICKS

	I.	J.	K.
Fe	1.65%	1.55%	1.50%
Fe_2O_3	0.70	0.92	0.63
Mn	1.08	1.08	1.02
SiO_2	30.69	33.28	34.87
Al_2O_3	15.04	14.16	15.37
CaO	44.65	43.81	41.17
MgO	4.01	3.45	4.06
S	1.13	1.29	1.16
Missing	1.05	0.46	0.22

The bricks thus made were of a pleasing bluish-gray color. The average weight was somewhat less than a sand-lime brick or a common clay brick of the same volume.

The Test Proper.

Method of carrying out tests. The results obtained and some conclusions to be drawn from these results.—A question of prime importance in regard to this proposed slag utilization is one that pertains to the quality of the slag-lime brick. As one familiar with the brick industry realizes, there are no standard specifications for testing bricks and likewise there are even a few, who ridicule the advisability of any specifications. For this investigation the brick testing specifications of Mr. A. V. Bleininger, Chief of the Ceramic Division of the U. S. Bureau of Mines, were employed. These specifications include compression tests on bricks free from moisture but otherwise untreated, compression tests on bricks saturated with water, compression tests on bricks which had been frozen while wet and subsequently dried, compression tests on bricks which had been subjected to fire and afterwards air-cooled, and likewise compression tests on bricks which had been subjected to fire and immediately quenched in water. They include a transverse test on bricks which had previously been subjected to the conditions set forth in the compression test and an absorption test on the bricks. A determination of the weight of each type of brick in pounds per cubic foot was also made.

The data on the I. J. K. series of slag-lime bricks or the bricks of Lot 3 are reproduced herewith and for comparison the variation from the standard set by Bleininger for standard building brick. Neither the fire nor freezing tests were made on this series.

Regarding the test as a whole it can be stated that comparison may now be made between the 14 sets of slag-lime brick which, as previously stated and explained are grouped into three lots, and various other forms of building bricks. All the tests here quoted were carried out under the author's direction in order that the results might be comparable. The various forms of commercial building bricks consisted of three different lots of sand-lime bricks, one of common clay brick, two of vitrified building brick, and one of repressed brick. The figures are given as an appendix in Tables I,

	Bleininger Standard.	Series of Slag-lime Bricks.		
		I.	J.	K.
Average weight of brick in pounds per cubic foot	.....	113	109	97
Transverse test on untreated brick. Modulus of rupture	425	565	441	383
Transverse test on brick saturated with water Modulus of rupture	325	500	429	443
Compression test on untreated brick. Strength in pounds per square inch	2500	4385	4026	4580
Compression test on brick saturated with water. Strength in pounds per square inch	2000	2970	2733	3179
Per cent of water absorbed	20.0	15.3	17.5	19.7

II, III, and IV. The results are shown graphically in Plates 1, 2, and 3.

The vitrified and repressed bricks are not of the same class as are the common clay, sand-lime, and slag-lime bricks but the results are included because of their interest. The direct and essential comparisons should be between a theoretical brick just within the bounds of specifications, a sand-lime brick, a common clay brick, and a slag-lime brick.

It is noted that in practically all cases the common clay brick is superior to both the sand-lime and slag-lime bricks. This is due to the fact that the clay from which these bricks were made is of as high a grade as any common building clay found in the United States. It is also noted that in practically all cases the sand-lime brick is superior to the slag-lime brick. In some measure this is due to the fact that the sand-lime bricks had been allowed to set for 6 months after their steam bath while the slag-lime bricks were but 6 weeks old. Another reason manifest for the poorer quality of the slag-lime bricks is because of the void question. Granulated slag—these bricks were made from granulated slag—contains about 52 per cent voids. Fine crushing, even to a point which permits all of the product to pass through an 80 mesh screen does not eliminate all of this trouble. A microscopical examination of the ground slag dust discloses the fact that much of the dust is a hollow spherical mass. This hollowness of the particle makes it structurally weak. Since this investigation was first performed the writer has been considering various means for meeting this

difficulty as efficiently and effectively as possible. He feels that were slowly cooled slag or slag cooled en masse used, instead of granulated, or water, or air cooled slag, there would be manifested a noticeable improvement in the quality of the brick. The results of the tests on the slag-lime bricks seem to bear the writer out in this theory. The materials in the bricks of Lot 3 were ground finer than the materials in the bricks of Lot 1 and Lot 2. In all cases Lot 3 bricks were superior. The constituents in the bricks in Lot 1 were pressed harder than the constituents in the bricks of Lot 2. In all essential places Lot 1 bricks are superior to Lot 2 bricks. Greater pressure and finer grinding have helped to remove the globular nature of the slag. Thus the better quality of one lot of slag-lime bricks over that of another is accounted for.

It is observable that the freezing test made practically no impression on any of the bricks. It is not believed that such a result was due to the fact that all the bricks tested were impregnable to such a test. It is felt that the test was not effective for it was not possible at the time the test was made to go below 20° F. whereas the standard temperature for the test is 15° F. After the freezing, each brick was put into a drying oven for the purpose of driving out all free moisture. Thus one would expect to get concordant results with those that were obtained on bricks free of moisture, but, in other respects, untreated. This was what proved to be the case.

It has been asserted that lime bond bricks have much better fire resisting properties than other types of building bricks. This may be true when the bricks are en masse. It was not true in this case when the bricks were subjected individually to a fire test. In all cases the lime bond bricks swelled badly, cracked, and spaulled, so that, in many cases, it was impossible to test them. This was particularly true of the slag-lime bricks.

It would be interesting to give the complete results of the absorption test. At the present writing, however, the only figures at hand are those which show the absorptive power of the various bricks at the end of 48 hours. Although the slag-lime bricks absorb a greater percentage of water than the other bricks, they can be made, and, in the cases of Lot 1 and Lot 3, were made to be within the specifications. Common clay bricks usually absorb their full amount of water within an hour. The lime bond bricks absorb

water much more slowly and gradually. This characteristic, many believe, is not for the best. Yet, it is true, that after 36 hours have elapsed no further absorption is noticeable in the bricks. In all cases when properly made, the maximum absorption of slag-lime bricks is under 20 per cent, the figure which is mentioned in the specifications.

The result of these tests indicated that the slag-lime bricks were inferior in every respect to the sand-lime bricks, and likewise that they were vastly inferior in every way to the red clay building brick found in the district where these tests were made. By inferior it is not meant to imply that the slag-lime brick was below standard specifications. For the most part it was not, and in those particular instances where it did not come up to the standard, the conditions which brought these defects about can be so remedied as to raise the standard of the brick up to and above the required amount.

With regard to the slag-lime bricks, however, it is not a question as to whether they can be made to pass requirements, so much as it is a question of a comparison of their quality with the quality of the bricks which are made in the same district. In the district where this particular test was made the building bricks are of an unusually high grade. They are of a high grade because the manufacturers have no difficulty in making a high grade brick, due, quite largely, if not altogether, to the fact that the clay with which the brick makers are working is, for the purpose, of as fine a quality as is found in the United States.

When one goes to other districts, however, conditions are found to be different. The bricks in the Philadelphia district, New York district, Chicago, and Detroit districts and in almost all parts of the South and West are known to be uniformly poor. This condition is due quite largely to the fact that the brick manufacturers do not have a very good quality of clay to start with. In such districts the slag-lime brick would have an excellent chance of surpassing in quality the grades of common clay building brick which are at present found on these markets. If there are blast furnaces in these districts finding difficulty in economically disposing of their slag, it impresses the writer that the conversion of their present waste product into a slag-lime brick would be a question worthy of careful consideration, in as much as slag-lime bricks can be made as cheaply as other kinds of bricks and like-

wise in as much as the quality of the resultant brick would be superior to that now on the market.

It is of course understood that it would not prove to be practicable to ship slag-lime bricks any great distance any more than it is practicable at the present time to ship the ordinary building brick a considerable distance. The question of freight rates enters into the commercial consideration of this problem and thus, to a certain extent, limits the range of this utilization.

Thus, one can see that the question of converting slag into slag-lime bricks is not one possessing uncertainties as to whether or not a slag-lime brick can be made. That question has been settled and slag-lime bricks can be made and produced as cheaply, it is believed, as any type of building brick at present on the market. It is not known whether or not the slag-lime brick will deteriorate under long service. This point can only be settled by a long time test. From the manufacturing standpoint it is a question of local consideration around the immediate vicinity of each blast furnace or group of blast furnaces. If the demand for bricks is large, if the grade of bricks at present made are poor because of an initial poor quality of clay, and if there is no ready outlet for the disposition of the slag, it would then behoove a blast furnace manager to consider seriously such a utilization.

TABLE I

Summary of Tests on Various Types of Building Bricks

Kind of Brick.	Type of Brick.	Transverse Test. Free from Moisture. Saturated with Water.				
		Weight of Brick per Cubic Foot in Pounds.	Modulus of Rupture.	Variance with Requirements in Per Cent	Modulus of Rupture.	Variance with Requirements in Per Cent
Sand lime.....	1	107.6	737	73.5	362	11.4
"	2	110.7	498	17.3	304	6.4
"	3	112.6	825	94.0	461	40.1
Average.....	....	110.3	687	61.6	376	15.6
Slag lime, Lot 1.....	A	109.6	357	16.0	273	16.1
" "	B	115.7	418	1.6	236	27.4
" "	C	111.9	413	2.8	252	22.7
" "	D	113.0	421	0.9	212	34.9
" "	E	111.7	448	5.4	243	25.2
" "	F	109.6				
Average.....	....	111.9	411	3.2	244	25.3
Slag lime, Lot 2.....	G	93.4	358	15.8	239	25.4
" "	H	96.8	351	17.4	221	32.0
Average.....	....	95.1	355	16.6	230	28.7
Slag lime, Lot 3.....	I	113.0	565	33.0	500	54.0
" "	J	109.0	441	4.0	429	32.0
" "	K	97.0	383	10.0	443	36.0
Average.....	....	106.0	463	9.0	457	41.0
Common clay.....	....	129.8	1242	192.3	1142	251.3
No. 1. Repressed.....	....	133.6	988	132.5	876	169.0
No. 2. Vitrified.....	....	134.9	1935	355.1	1456	348.0
No. 1. "	....	141.4	1639	333.0	1413	335.0

TABLE II

Summary of Tests on Various Types of Building Bricks

Kind of Brick.	Type of Brick.	Transverse Test.					
		Saturated with Water Frozen. Then Dried.		Originally Brick Subjected to Fire.			
				Air Cooled.		Water Quenched.	
		Modulus of Rupture.	Variance with Requirements in Per Cent.	Modulus of Rupture.	Variance with Requirements in Per Cent.	Modulus of Rupture.	Variance with Requirements in Per Cent.
Sand lime	1	618	125.0	No fire t	ests were	made on	this lot
"	2	607	121.8	"	"	"	"
"	3	762	177.0	"	"	"	"
Average	. . . .	662	141.2	"	"	"	"
Slag lime, Lot 1 . . .	A	419	52.4	"	"	"	"
" " . . .	B	514	86.9	"	"	"	"
" " . . .	C	434	57.8	"	"	"	"
" " . . .	D	268	2.5	"	"	"	"
" " . . .	E	314	14.2	"	"	"	"
" " . . .	F						
Average	. . . .	390	41.6	"	"	"	"
Slag lime, Lot 2 . . .	G	225	18.2	"	"	"	"
" " . . .	H	266	3.3	"	"	"	"
Average	. . . .	246	10.7	"	"	"	"
Slag lime, Lot 3 . . .	I	No freez	ing or fir	e tests w	ere made	on this lo	t of brick
" " . . .	J	"	"	"	"	"	"
" " . . .	K	"	"	"	"	"	"
Average							
Common clay	. . . .	1244	352.0	182	263	184	266
No. 1. Repressed . .	. . . .	1254	356.8	310	520	310	520
No. 2. Vitrified	. . . .	1806	356.8	85	70	89	78
No. 1. "	. . . .	1829	565.0	310	520	310	520

TABLE III

Summary of Tests on Various Types of Building Bricks

Kind of Brick.	Type of Brick.	Compression Test. (Expressed in Pounds per Square Inch.)					
		Free from Moisture.		Saturated with Water.		Saturated with Water Frozen. Then Dried.	
		Crushing Strength.	Variance with Requirements in Per Cent.	Crushing Strength.	Variance with Requirements in Per Cent.	Crushing Strength.	Variance with Requirements in Per Cent.
Sand lime	1	9083	262	5563	178	8738	385
"	2	7158	187	4988	150	6948	286
"	3	9402	276	7025	251	10252	470
Average	....	8548	242	5859	193	8646	380
Slag lime, Lot 1	A	3027	21	2171	8	3149	75
" "	B	3803	52	3516	76	4737	163
" "	C	3754	50	2662	33	3324	85
" "	D	2454	2	1960	2	2556	42
" "	E	2858	14	1632	18	2866	59
" "	F	3463	39				
Average	....	3227	29	2388	19	3346	85
Slag lime, Lot 2	G	2203	12	1475	26	2362	31
" "	H	2104	16	1366	32	2512	40
Average	....	2154	14	1421	29	2437	35
Slag lime, Lot 3	I	4385	75	2970	49		
" "	J	4026	61	2733	37	No freezing test	
" "	K	4580	83	3179	59	made on this lot	
Average	....	4380	73	2961	48		
Common clay	....	7043	182	8197	310	6522	264
No. 1. Repressed	....	8567	247	7680	284	8573	376
No. 2. Vitrified	....	11165	347	8197	310	10608	489
No. 1. "	....	11138	345	8498	325	11366	531

TABLE IV

Summary of Tests on Various Types of Building Bricks

Kind of Brick.	Type of Brick.	Compression Test. (Expressed in Pounds per Square Inch.) Originally Brick Subjected to Fire.				Absorption Test.	
		Air Cooled.		Water Quenched.			
		Crushing Strength.	Variance with Requirements in Per Cent.	Crushing Strength.	Variance with Requirements in Per Cent.	Total Absorption in Per Cent.	Variance with Requirements in Per Cent.
Sand lime.....	1	3362	87	4488	149	14.5	27
"	2	2484	38	Broke	.....	13.1	34
"	3	3395	89	"	.....	13.9	30
Average.....	....	3080	71	1496	17	13.8	30
Slag lime, Lot 1...	A	1535	15	3126	72	18.9	5.5
" " ...	B	2972	65	4045	124	16.7	16
" " ...	C	2102	17	3992	122	16.0	20
" " ...	D	Broke	.....	Broke	.....	16.7	16
" " ...	E	"	.....	"	.....	18.6	7
" " ...	F						
Average.....	....	1322	26.5	2233	24	17.4	12.9
Slag lime, Lot 2...	G	1472	18	Broke	.....	25.2	26
" " ...	H	1326	26	"	.....	24.0	20
Average.....	....	1399	22	"	.....	24.6	23
Slag lime, Lot 3...	I	.....	.....	.....	.....	15.3	24
" " ...	J	.....	.....	.....	.....	17.5	12
" " ...	K	.....	.....	.....	.....	19.7	2
Average.....	....	.....	.....	.....	.....	.....	13
Common clay.....	....	5850	225	6297	243	8.7	57
No. 1. Repressed..	....	8653	380	7905	335	6.2	69
No. 2. Vitrified....	....	9095	411	8882	401	1.1	94
No. 1. "	....	10399	477	11279	526	1.4	94

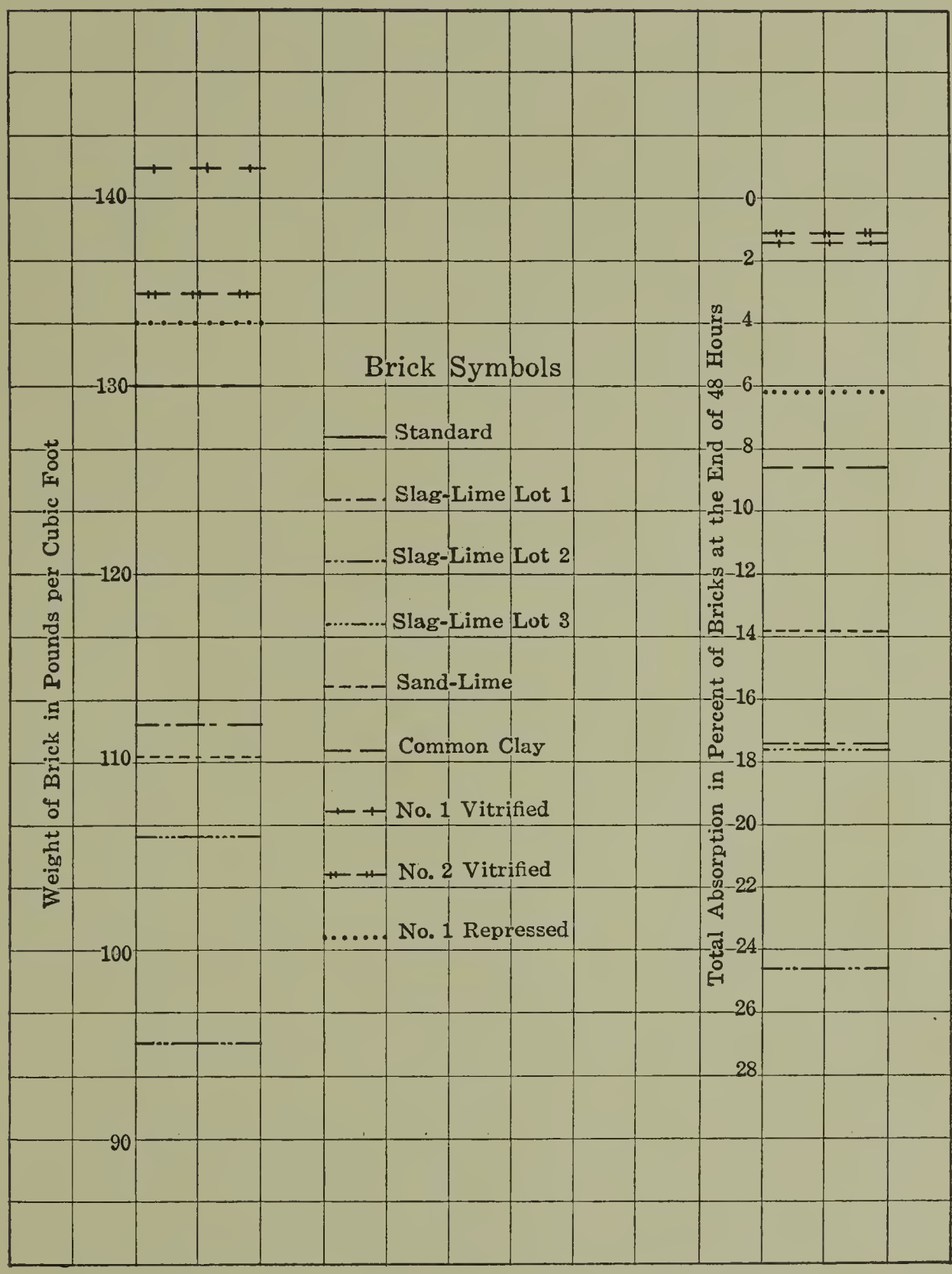


PLATE I.—Weight and Absorption Tests on Building Bricks.

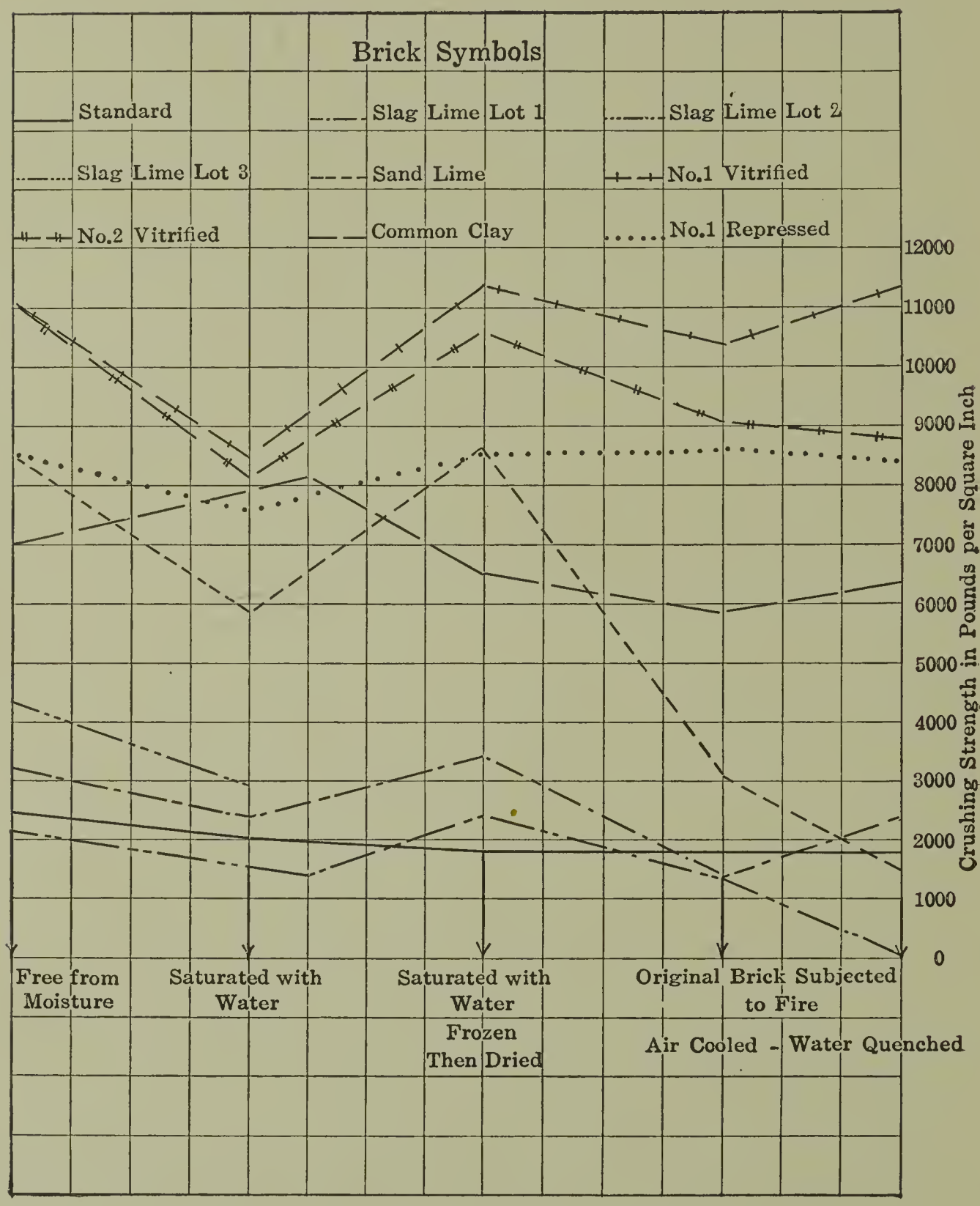


PLATE II.—Compression Tests on Building Bricks.

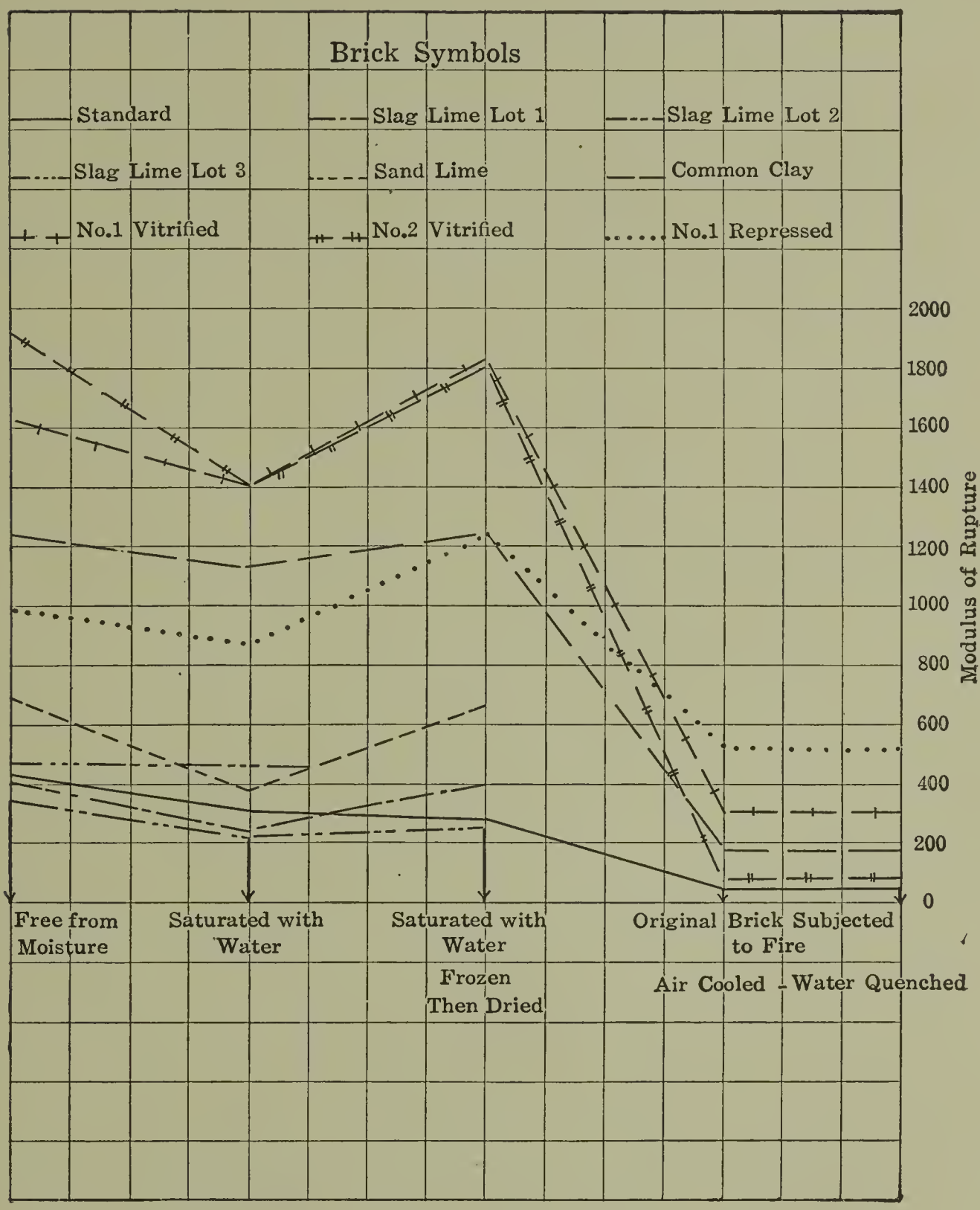


PLATE III.—Transverse Tests on Building Bricks.

TECHNICAL ACCOUNTING AND CHEMICAL CONTROL IN SUGAR MANUFACTURE

By DAVID L. DAVOLL, Jr.

Read at the Detroit Meeting, Dec. 6, 1912.

Introduction.

For the technical supervision of the manufacture of sugar, whether from beet or cane or whether the purpose be to make raw sugar or refined granulated, the activities of the chemist may well be directed along three lines of endeavor.

First, the sampling and analysis of all raw material such as coal, coke and limestone or lime. Almost without exception in the beet industry and in exceptional cases with the cane, this includes the daily systematic valuation of beets and sugar-cane deliveries for purposes of purchase. Further it will include the analysis of field samples to determine maturity of crop in general and the effect of certain influencing factors in particular districts.

Second, the frequent and rapid testing of initial material, intermediate products at the several "stations" of manufacture and the final products, together constituting what is known as "chemical control."

Third, the keeping of the sugar account and the daily calculation of the efficiency of the various pieces of machinery and of the several intermediate processes of manufacture.

Chemical control is essentially diagnostic in character and takes advantage of the knowledge we have of how the sugar-bearing material should behave at the "stations" as now interpreted through chemical tests and of what constitutes recognized, unavoidable losses, to put in the hands of the foremen in charge the proper data.

Successful sugar accounting calls for good organization first

and conscientious, well-trained chemists to carry it out. Success or failure depends upon the presence or absence of suitable conveniences, accurate factory weights and measures, calibration of utensils and the exercise of great care in the sampling, subsampling, compositing and preserving.

In the beet industry the polarization is practically the true sucrose (with raffinose absent) while in the cane industry the polarization is never the true sucrose but is the algebraic sum of the several optical activities, of all the participating bodies, not removable by lead acetate. Therefore, in this article the term polarization or "sugar" signifies the polariscopic reading when reduced to terms of 26 grams of material in 100 metric cubic centimeters. Sucrose refers to the Clerget figure.

A few terms peculiar to the cane industry, may well be defined here.

Normal Juice. Strictly speaking this is the whole juice of the cane as it exists in the tissues, or the combined juice of all the mill units when milling without the application of water of saturation. It still has considerable significance in cane work but none in the beet. It usually runs from 0.1 to 0.7 degree Brix lower than the so-called First Mill Juice; it is also lower in purity. Owing to its variability under changing conditions it should be determined by actual run, without water, at stated intervals.

It does not appear that a proper figure can be obtained in less than ½ hour's run. Its Brix, taken in conjunction with that of the mill raw juice, is used in calculating the extent of the dilution due to the water of saturation.

Mill Extraction. The percentage of the sugar in the cane that has been removed by the milling process.

Retention. The amount of sugar in the form of commercial sugar, expressed in terms of percentage of the sugar obtained in the milling process.

Total Efficiency. The total sugar in the form of commercial sugar expressed in terms of percentage of the sugar in the cane. It is the product obtained by multiplying the extraction by the retention.

Blanc. A product of vacuum-pan boiling upon very low products variously termed "filete" and "string-proof." It is not boiled to grain, but is made very concentrated and the density judged

by pulling a small quantity out to form a "string" or rolling a small ball in cold water and noting the hardness.

It is the final boiling in all houses not provided with crystallizers, the product being set aside in storage for slow cooling and crystallization, for from two to three weeks or more.

A very brief account of the processes of sugar manufacture may assist in making clear the purpose of chemical control and scheme of sugar accounting. United States beet and Cuban raw sugars only are considered.

Beet Sugar.

The factories of the United States make standard white granulated sugar in one operation from the sugar beet, which has a sugar content of from 14 per cent to 23 per cent average for the entire crop or "campaign," according to territory, season and seed pedigree.

1. *Diffusion Juice.* The beets are washed and then sliced into strips 3½ to 7 mm. wide and 2–4 mm. thick, with cross-section V-shaped and the sugar extracted by a highly developed process of diffusion, at definite temperatures, leaving behind the "pulp," of sugar content 0.25–0.30 per cent. There results a very clean, but dark colored liquor, warm to hot, of density 0.8–0.9 that of the original juice of the cossettes (sliced beets), which goes to duplicate tanks for measurement, after which it is forced by centrifugal pumps to tall, closed tanks.

2. *Liming and Carbonitation.* Lime to the extent of 2½ per cent to 3 per cent of the beets or lime-saccharate as produced, both in the form of "milk" of 17° Bé., is added, while simultaneously the mass is brought to a definite, high temperature by injected steam and carbonic acid from the lime kilns is pumped in. At the precise moment of granulation of the precipitate of $CaCO_3$, etc., judged wholly by the eye and corresponding to 0.1–0.14 per cent CaO alkalinity, the mass is released to the pumps which serve the set No. 1 filter presses.

3. *The Filtrations.* After issuing from the first set of presses under 2½ atmospheres of pressure in form of a brilliant, pale straw-colored liquor, it goes to a second set of tanks for further liming and carbonitation, where the alkalinity is brought to 0.015–0.025 per cent CaO. It is then passed through a second set of filter

presses and then "sulphured." The presses are clothed with duck or jute or both. The press cakes are washed with hot water (condensed) to a definite sugar content, set No. 1 to 1 per cent, set No. 2 to 0.5 per cent, judged by applying a suitable hydrometer to the issuing "sweet waters."

4. *"Sulphur Station"* No. 1. Bleaching by means of SO_2 gas takes place here, reducing the reaction to faint alkalinity, neutrality or faint acidity as occasion demands, but always to a definite figure. The process is perhaps invariably a continuous one.

5. *Gravity Filters* No. 1. Passing through these is preparation for evaporation under multiple-effect system.

6. *Concentration.* By means of quadruple or quintuple effect the density is raised to 60° Brix, some ammonia is given off, lowering of alkalinity in the absolute but rising as result of concentration; some precipitate forms. "Thick juice" results.

The work is now in the "boiling" house.

7. *"Sulphur Station"* No. 2, or *"Blow-ups."* The reaction is here brought to the desired point, either faintly alkaline or acid and by careful test, in accordance with a figure that has been found to give the best results at that factory and particular district and often influenced by the season: 0.02 per cent CaO (basis) is seldom exceeded either way. Acidity may be aided by phosphoric acid; alkalinity induced by lime or soda ash. The thick juice, like all products passing this station, is here "blown up" with injected steam and reduced to uniform Brix, quite generally 60°, to dissolve any grain, aid filtration and promote good boiling in the pan.

8. *Gravity Filters* No. 2. These serve all products in the intricate but systematic work of the "boiling" house such as thick juice, high and low wash and greens and melted sugar, removing all suspended matter in preparation for the pan-service tanks.

9. *The Massecuite.* This is formed in a vacuum pan working under 26–28 inches of vacuum and in masses of many tons.

The boiling to grain is carried out purely as an art by men who make it a profession. Of course, it is based entirely upon well-known scientific principles. It is essentially crystallization-in-motion, during which the product, constantly augmented in quantity by fresh injections of liquor, is resolved into grains of sugar and a more or less de-sugared mother liquor.

The final result of the process is judged upon the so-called purity test. The initial product from the beets generally carries a purity of 88; as the sugar crystallizes and is removed by the centrifugals the purity ratio necessarily lowers, thus becoming a measure of efficiency and of paramount importance.

The first boiling, followed by hot turbinating, gives, upon washing or "covering" with diluted liquors, white refined sugar, a "wash" of 90–92 purity and a mother liquor of 78–80 purity. A second boiling to grain, massecuite having 78–80 purity, discharging while hot to the crystallizers, cooling there with the aid of a helix and water jacket for about 3 days and then centrifugating, gives a yellow sugar and an exhausted molasses, purity 58–60. The yellow sugar is redissolved and boiled, variously combined, to white refined sugar.

If it is the intention to recover still more sugar from the molasses so reduced in purity that it will yield no more sugar by crystallization, the sugar may be precipitated by a large excess of one of the oxids of the alkaline earths.

In the United States lime oxid is used, in absolutely anhydrous and impalpable powder. Tricalcium saccharate results, later soluble in the saccharine juice to monocalcium saccharate.

10. *The Coolers.* Under agitation the powdered lime is slowly sifted into the molasses previously reduced to 12–14° Brix, kept cool at a definite temperature meanwhile, until the density of a filtered sample indicates 6–7° Brix. It is then filter-pressed as quickly as possible.

11. *Saccharate Presses.* The ordinary Kroog type of press produces 40 cakes of 1 inch thickness; the saccharate presses produce a much thicker cake, usually 2½ inches thick; they fill and wash readily. The product from the coolers is here separated into the saccharate of 86–98 purity and a mother liquor known as "press waste water" of 6–7° Brix and 10–20° purity, all from a 58–62° purity molasses. The washing with cold water alone or combined with its own higher "sweet waters" is continued until a liquor having a Brix of 2½ to 3° results and the purity of the last runnings mounts to 15–30. The total product of the washing is known as "wash water."

12. *Saccharate and Saccharate Milk.* The saccharate press cake is transported to the liming and carbonating station of the

main factory process, in the form of milk, being discharged directly into tanks provided with stirrers, where it is incorporated with liquors brought thither from various parts of the factory and bearing from known small amounts of sugar to mere traces.

A separate sugar account is required for the saccharate process and the yield should be about 67 per cent of the sugar charged to it, in the form of refined white.

Cane Sugar.

Raw sugar factories dealing with cane aim to produce a sugar that will keep during storage and transportation, of a sugar content that will bring the highest price for the total sugar output and to get as high a yield as can be proved to be economical in the final summing up of all the conditions.

It is generally conceded that an even 96° Ventzke polarization at the port of entry brings the highest profit. It is very probable that all other grades will soon be suppressed.

1. *The Milling.* Heavy iron mills replace the diffusion battery of the beet-sugar process. The installation generally consists of three units (individual mills) placed in tandem and composed of three rolls each; there are intervening conveyors and the whole train is preceded by a crusher of two rolls.

The crusher serves to break the outer rind and the nodes, liberating at the same time considerable juice which flows to the bed plate of the first unit. The rolls are ponderous; 7 feet long by 3 feet in diameter may be taken as a type.

The cane gets two compressions in passing through each unit, being sustained by the "turn bar" as it issues from between the cane roll and the top roll and passed on to be caught by the bagasse roll and the top roll for the still closer compression.

The cane, not being laden with molasses-forming salts to the great extent that the beet is, the rupture of the cells is not attended with disastrous consequences. Naturally the ratio between the fiber of the cane entering any given unit of the tandem and that of the bagasse issuing therefrom, is a measure of the efficiency with respect to the amount of liquid expressed. Where water of saturation is applied, hot or cold, it is generally sprayed forcibly upon the bagasse as it issues from the unit next the last, at the point of immediate

release from pressure; the imbibition of the thin juices is the further development of the water treatment and is simple and effective. As high as five units and a crusher or in all, seventeen rolls have been employed in one tandem.

The juices from the first and second units only enter the process of manufacture, *i. e.,* under a system of combined saturation and imbibition, and, united, constitute the mill raw juice.

2. *Mill Raw Juice.* Sometimes called diluted juice.

With respect to its purity it is considered to represent the original juice of the tissues of the cane.

It is either at factory temperature or a trifle above it, depending upon the saturation water temperature; it is charged with air, turbid from suspended albuminous matter, wax, insoluble salts, clay, and fiber—this even after being strained through copper or brass sieves of 15–19 perforations per linear inch.

It is pumped to tanks for measurements or weighing and is then limed. Where heavy liming with carbonitation is not practised (and I know of no factory in Cuba doing this) the juice is only neutralized.

3. *Liming or "Tempering" the Juice.* This is generally accomplished in sets of three large tanks per tandem, one filling, one under treatment and one discharging. Repeated trials upon different sized tanks have shown a size corresponding to three hectoliters per ton of cane per hour to be advantageous.

Chemists carry the reaction of the juice generally about neutral to litmus paper. The subject can be said to have been scarcely attacked from the quantitative standpoint.

Continuous liming is practised in some factories.

Lime and heat form clear juice and cachaza or "scum."

After liming, the juice is passed through heaters where the product may be even superheated if desired, depending upon whether it is desired to eliminate all the air by a "flashing" operation before complete settling. If the air is not eliminated a thick scum rises to the top at 95° C. called "blanket," a small portion sinks to the bottom, while the separation is being effected in so-called defecators varying in size from 35–100 hectoliters, net; the time for making one complete round of the defecators will be 60 minutes but capacity should be had for 90 minutes, to allow for irregular liming; 15 per cent of the time will be consumed in filling, emptying and cleaning.

Continuous settling is effected in the Deming process and in the Hatton defecators.

The slightly opalescent, straw-colored juice is generally run, without filtration, merely decantation continuous or interrupted, directly to the multiple effect.

It should be passed through fiber or gravity filters if for nothing more than to catch much cachaza that slips into the process intermittently. The decanted cachaza is washed by decantation in small tanks and then sent to frame presses for compression and sometimes washing. Evaporation is carried to 55° Brix in a cane house, to facilitate (1) settling and (2) avoidance of false grain.

The boiling of the meladura to grained massecuite is similar in principle to that carried out in a beet factory.

Cane products grain with great facility, while beet products sometimes present great difficulty, conditions brought about by the variance in the character of the non-sugars, purity remaining the same.

Generally three grades of grained massecuites are boiled where crystallizers have been installed, all upon a nucleus of original meladura which ranges in purity from 80–92, according to district and time of season. *First massecuite,* purity 80–84, yielding a sugar polarizing 97–98° Ventzke and a corresponding green syrup or molasses of purity 60–64. *Second massecuite,* purity, 70–74; corresponding molasses, centrifugated hot, 48–54; centrifugated after limited cooling in motion, purity 40–46; resulting sugar, washed by water or liquors to 96° V. *Third massecuite,* purity 58–63; corresponding molasses (final product), purity 30–35; resulting sugar polarization depending upon treatment. This last massecuite, when at 35–40° C. and 4–5 days old in crystallizers, is generally centrifugated and the untreated sugar discharged into a mixer where high-grade molasses is incorporated with it and it is again centrifugated and washed to the degree desired, generally 96. This process is styled "mingling."

In factories not provided with crystallizers (which keep the grain in motion) the exhaustion of the product when it reaches the purity 48–54 must be accomplished "at rest," which is brought about by discharging the final boiling, boiled "blanc" to a Brix of from 88–91, according to conditions, into small iron wagons or into large tanks where it is allowed to cool quietly and crystallize spontaneously for from 12–21 days or longer.

The Control

SAMPLING AND ANALYSIS

The Cane. Determine fiber and sugar once each factory day.

Sampling. Every hour, four representative canes are to be selected under the chemist's supervision, as they pass from cars, wagons or hopper to the first unit of the tandem. Reserve in a cool, shaded place. If the factory runs six hours or less, prepare the whole sample; if more, subsample to 24 canes as follows: Sort into three piles, one containing the pieces bearing evidence of having been cut in the fields next to the root; one, pieces cut from the

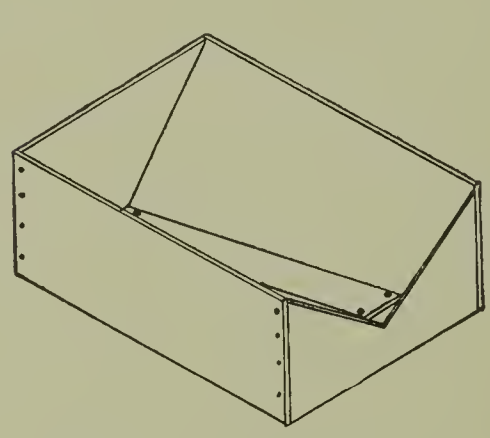


Fig. 1.—Hopper for Chopping Cane.

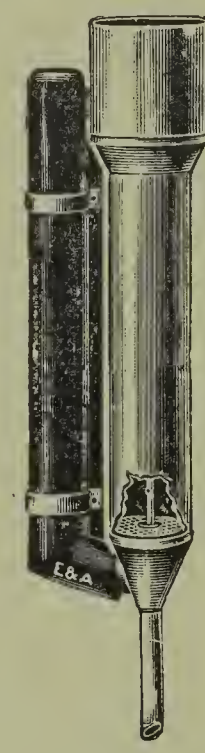


Fig. 2.—Continuous Extractor.

middle; one, pieces bearing base of the "cogollo" or top of the cane. Take, impartially, eight pieces from each pile and cut into transverse slices 1/8–1/16 inch thick by means of a Pellet cane cutter, feeding the tops to the machine first and rejecting no odd ends. Allow the slices to fall into a galvanized iron box large enough to hold the entire sample; throw upon a large, clean piece of enameled cloth, mix well and quickly subsample by "coning and leveling" until about a liter is obtained; weigh this carefully. Chop in a hopper (see Fig. 1) resting upon a clean piece of enameled cloth, with a heavy

cane knife (calaboso) when finished, brush up all pieces that may have fallen upon the cloth and again weigh.

Correct for loss by drying out during chopping. Hopper must be employed for cane exclusively.

Sugar Determination. Lightly pack 52 grams in a continuous extractor[1] and slowly pour through the mass sufficient 40 per cent alcohol to fill the 4-oz. Adam's flask two thirds full; maintain the flask contents faintly alkaline with basic lead acetate; extract for three-fourths hour or for such time as experience shows, under working conditions, is sufficient to extract the sugar to 0.05 per cent limit using a perforated asbestos plate and low flame. Place the flask upon the water bath and expel the alcohol; rinse into a 100 cc. flask, add sufficient basic lead acetate, fill to the mark, filter and polarize. Reading divided by 2 equals sugar.

Fiber Determination. Lightly pack 52 grams in the continuous extractor, allow cool or tepid water to run slowly through during 1–2 hours; then, slowly, about a liter of water heated to 60° C.; connect the flask containing 40 per cent alcohol and extract for three-fourths hour; withdraw dregs by means of the rod attached to the bottom sieve diaphragm to a drying dish; dry for 2–4 hours to constant weight at 105–110° C. Calculate fiber. To dry cane or bagasse fiber, employ shallow oblong tin trays, 4″ by 5″ and 1⅝″ deep, bottom consisting of copper cloth of 80–100 meshes per linear inch.

THE BAGASSE. Sugar may be determined every 2 hours, fiber once a factory day. Well ground bagasse (modern milling) may be taken by the handful as it rises from the mill-boot of the discharge conveyor and tightly pressed into the sample can, which may be 2 feet deep by 1 foot diameter. Fifteen minutes intermittent sampling should fill the can and there should be the minimum delay in preparation for analysis.

Coarse bagasse (such as that of the first, second or even third

[1] This extractor is made of nickeled copper and is very durable; vapor pipe insulated, thus promoting rapid exhaustion of contents besides serving as a handle; it is suitable for use with cossettes, "pulp," drug and material of many kinds calling for restricted quantity of solvent.

Especially designed to eliminate "bumping" entirely. An easily removable screen diaphragm retains the material in place, which, after extraction, may be quantitatively removed for drying and weighing. Made by Eimer & Amend, New York. Standard size 11 cubic inches net capacity.

unit of the tandem on old-style milling) should be taken from its particular conveyor, *clear across* the blanket and amount to at least 1½ cu. ft. in volume.

Throw the can contents to the floor upon a large sheet of enamelled cloth, tearing coarse pieces apart by hand. If still warm (hot saturation) cover lightly with a second sheet of cloth for a

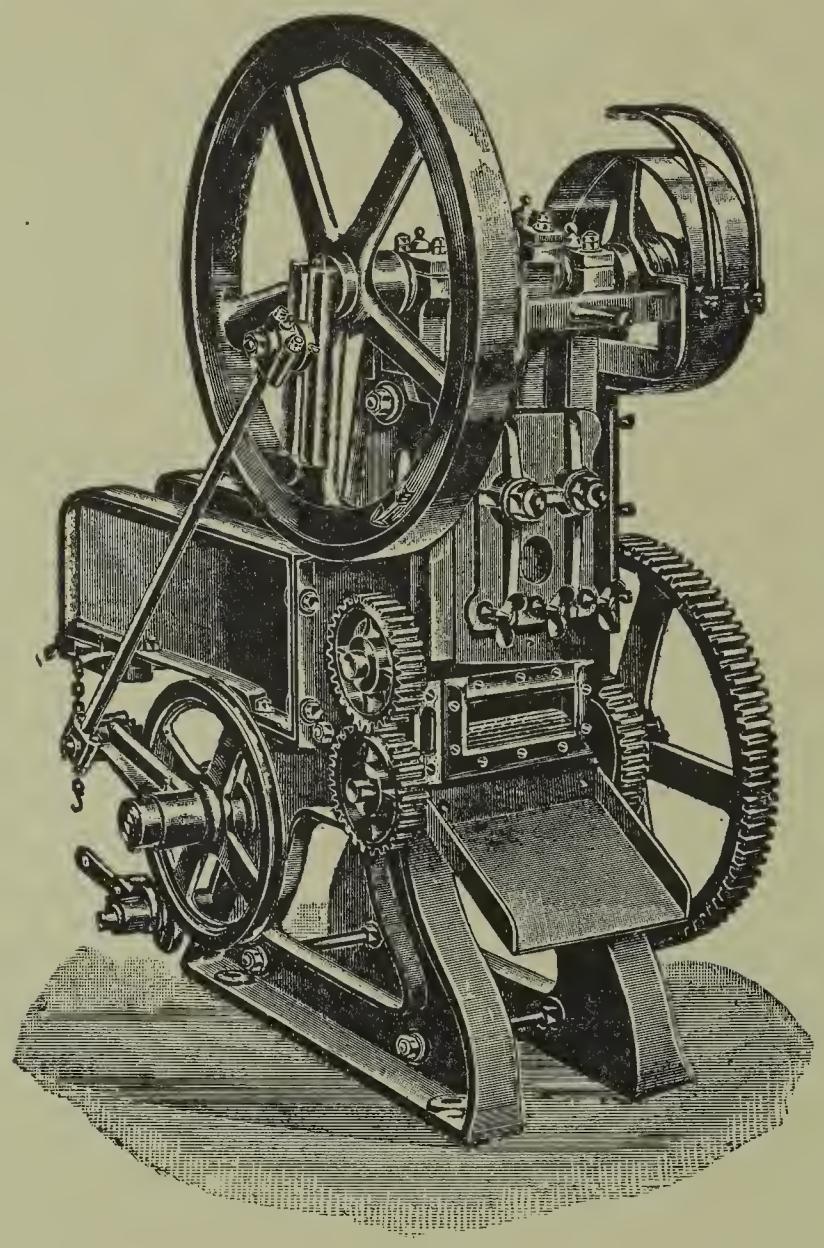


FIG. 3.—Bagasse Cutting Machine.

few minutes. Rapidly mix by stirring and rolling, exposing the minimum surface and pile in a cone; level from the apex outward to a truncated cone and withdraw a wedge-shaped sample, in size proportionate to the coarseness of the bagasse. Cut up the whole sample and mix. A fully satisfactory machine for quickly reducing large amounts of bagasse to fine "sawdust," is that made by Boot and Krantz, The Hague, Holland. See Fig. 3.

Polarization. This is performed as under cane, with the alkalinity maintained with a 5 per cent solution Na_2CO_3.

Fiber Determination. This necessitates a very finely divided material. The Hawaiian Sugar Chemists' Association defines fiber as "the total insoluble solids," water being the solvent.

Quantitatively remove the residue left after extraction of the sugar to a drying tray and dry for about an hour, then transfer to a large, loosely covered container; do this on every sample of the factory day. At the close of the day there should be, in the container, the practically dry residues of 12 normal weights. Mix well, weigh the accumulated samples, take one-twelfth, place in a drying tray and dry for 2–3 hours or to constant weight. Divide the weight by 26, multiply by 100, result is percentage fiber in bagasse.

If 10 samples only have been extracted, take one-tenth, etc.

Alternate Sugar Method. Use that as adopted by the Hawaiian Chemists' Association, 1910, *Bull.* 32, Agricultural and Chemical Series, Experiment Station, Hawaiian Sugar Planters' Association, by W. S. Norris.

Moisture. As this serves to judge of the tax laid upon the furnaces by reason of the water to be evaporated; calculate by "difference."

Mill Raw Juice corresponds to the diffusion juice of the beet industry. It is the main basis of sugar accounting and great pains should be taken to make it fully representative of the work.

Sampling. The following method by means of a thin, rapidly-running stream has been found to be representative.

Modern mills, in general, discharge the mill raw juice from the free end of a pipe into a small reserve or over-flow tank, which in turn serves the measuring or weighing tanks proper.

At a point a short distance below the level of the discharge (in order to secure a slight "head") the main discharge pipe is tapped by a half-inch pipe in such a manner that a small quantity of juice continuously finds the way to its destination, the reserve tank, through it. A small copper wire, preferably not over 4 inches long, may now be attached to the end of the half-inch pipe and a thin stream of juice diverted so as to discharge through a hole in the side of a covered, 2-gallon pail. Another location for this half-inch pipe, not quite so advantageous, is in the same main raw juice line but close to the pump, returning the diverted

portion of juice to the pump-tank and interposing the copper wire in the stream, as described. This latter arrangement reduces the pump efficiency about 1 per cent in a 1000-ton factory. The half-inch pipe should have but one cock, and that next the open end, to allow for closing when the pump is not operating.

The collection of samples is apt to be a weak point, on account of placing too much reliance upon a messenger. It should be reduced to a simple system, proper conveniences supplied, and closely supervised.

In tropical work, evaporation is rapid and the air is full of spores. All samples should be removed from the factory at frequent intervals and let the chemist either analyze them at once or properly preserve them. The messenger may collect the following samples every two hours. Mill raw juice, first mill juice, last mill juice, residual juice, evaporator thin juice and meladura. Assuming that these are already discharging into their containers there will be needed for substitution six other clean and dry sample pails containing sufficient formaline. Not all of the sample collected can be or should be taken to the laboratory—after mixing, the greater bulk will be at once returned to the process of manufacture. For this purpose, separate clean and dry metal paddles will be needed for stirring. A tray of light material holding six, liter Mason jars, plainly labelled and with screw tops will be needed for receiving the sub-samples.

Analysis. Strain the sample into a clean, well-rinsed Mason jar and let stand until the foam has risen; carefully remove the same with a teaspoon.

A. The Brix. Slowly fill a tall cylinder by pouring down the side; carefully insert the hydrometer, let stand until it has come to permanent rest, then read and note the temperature; correct for hydrometer error, if any and to standard temperature.

B. The Polarization. Slowly fill a 100–110 cc. flask to lower mark, clearing up any uncertain meniscus with a drop of ether, run in the prescribed amount of basic lead acetate and fill to the upper mark with water; shake thoroughly, filter and polarize, employing the Schmitz table for sugar percentage. If the analyses must be made at longer intervals, composite as follows: Prepare as above for "A" and "B;" instead of proceeding with "A," pour 200 cc. into the compositing jar containing the proper amount of

formaline; for Brix: proceed with "B" as far as filtration, then pour the whole contents of the flask into the compositing jar for polarization.

When making the determination, thoroughly mix the contents of each compositing jar, proceeding as under "A" for Brix. Filter, polarize and consult Schmitz's table for sugar percentage.

C. Set aside in a suitable jar a portion of the unfiltered "leaded" solution for the daily composite determination of sucrose by the Herzfeld-Clerget method and invert sugar.

When making these last two determinations, add sufficient acetic acid to transform all basic lead acetate into neutral or slightly acid condition, correcting for increased volume in final calculation.

First Mill Juice. *Sampling.* This must be representative of juices coming from combined crusher and first unit of the tandem. There is no agreement in either Brix or sugar content nor in the quantity of the juices falling from (1) the crusher, (2) the cane roll, (3) the bagasse roll; hence the logical place for continuously and systematically drawing this sample is in the trough conveying it to the mill raw juice pump. Fig. 4 shows a successful device for taking this sample.[1]

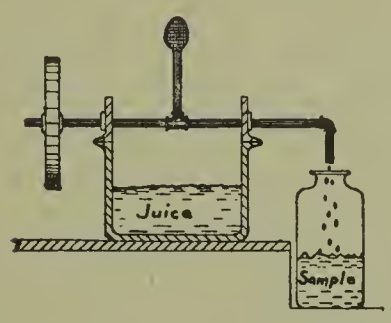


Fig. 4.—Juice Sampler.

An objection to this will sometimes be that water used to cool the mill bearings finds its way into this sample, the effect being to raise the figure for extraction.

The trough sample then becomes useless and a second sample should be taken by placing a suitable pail beneath the cane roll of the first unit at a point where tests show that the Brix and sugar content correspond to those of the trough sample when not contaminated by water. This pail holds about 8 liters and has a cover slightly inclined; holes are made near its apex, from the inside outward, sufficient in number to about half fill the pail in 2 hours.

Analyze as under mill raw juice, "A" and "B" only.

Third Mill Juice. The sample, where saturation is practised,

[1] The "spoon" sampler consists of a large spoon having a hollow handle of copper communicating with a hollow shaft of small piping and discharging through an elbow into a pail charged with formaline. The shaft, driven from the mill-roll should make about 8 r. p. m. The spoon is covered with fine screen to keep out "trash."

is taken from its pump discharge pipe as described under mill raw juice. Where it is not, the spoon sample may be used in the trough. For analysis, see under mill raw juice, A and B only.

Residual Juice. This is the drip from the bagasse roll of the last unit of the tandem and should be taken continuously, as under first mill juice and analyzed as under raw mill juice, "A" and "B."

Evaporator Thick Juice. The Brix of each tank filled should be taken. Where fluctuation in purity is considerable, as between 70 and 85 for instance, facilities should be at hand for rapid determination of purity. For this and similar work the author has introduced the Pellet continuous polarizing tube.

Filter Press Cake. The loss from this source is seldom correctly determined. At reasonable intervals the laboratory messenger should go to the trucks located below the presses and as a press is dumped, break off small pieces from a dozen large cakes; if semi-fluid a cup should be used. Analyze in the usual way but substitute 25 grams for a normal weight to allow for insoluble material. Periodically the weight of the press cakes should be determined.

The Sugars. Large centrales employ fans for cooling and rendering the sugar more uniform. Where not used, the net weight per sack must be verified when cold and finally loaded. The weight is still held by custom at 325 Spanish pounds of 460 grams each.

Sampling. A clean and dry galvanized iron box of about 1 cu. ft. capacity and having a funnel-shaped hopper in the cover, is placed at the sugar scales, to be changed once every 6 hours.

From every bag in five, and before adjusting the weight with sugar from the storage bin, the truckman will transfer a pinch of sugar from bag to sample box. As the sample is taken away, the serial number of bags filled will be recorded, in order to arrive at the number of bags represented, in proportion to which the polarization is to be adjusted in taking off averages.

Preparation. The sugar is poured upon a plate of glass, all sticks and foreign matter removed and thoroughly mixed with a clean steel spatula. Lumps are reduced with a porcelain roller and incorporated with the rest of the sample. Polarize at once. Determine moisture once a day. Composite a small portion from each sample for the semi-monthly chemical statement upon which is

to be determined, (1) polarization, (2) true sucrose, (3) dry substance, net (4) invert sugar (5) ash, (6) total dirt, (7) ash in total dirt.

When the sugar contains over 1 per cent moisture, the sample for compositing is to be dried in a water-bath oven for a short time, later correcting the final analytical data back to the basis of the average of the daily polarizations upon the fresh sample.

Polarization. A normal weight is placed in a funnel and washed into a 100 cc. flask with 50 cc. of water, completely dissolved by rotating, then clarified by lead-*b*-acetate solution and 2 cc. of alumina cream. As a rule not over 1 cc. of lead solution is needed for high-grade centrifugal (96) sugars and from 2–6 cc. for molasses sugars (80–90). Use the minimum quantity necessary for clarification. After the lead and cream are in, allow air bubbles to rise and complete volume to 100 cc. Mix and filter in a carefully covered funnel, discarding the first runnings. Endeavor to polarize at the temperature of dilution.

THE MASSECUITE. Applicable to either grained or blanc strikes.

Measuring. Massecuite intended for the crystallizers should be measured after being placed therein, at the moment of entering and for every strike, the same applies to sugar wagons or tanks. The volume of material subject to crystallization in motion for long or short periods or "at rest" for many days, should be known, as an important step in control.

An accurate account should be kept of the movement in and out, so that at any time, by consulting the records, a balance can be struck of the exact amount in stock.

Sampling. Take a portion equal to 2 liters from at least three places as the mass is struck from the vacuum pans, *viz.*, after it is running well, in the middle of the flow and toward the end.

Analysis. At the laboratory the following tests are made. (1) Purity of the mother liquor, (2) brix by double dilution, (3) polarization.

Purity of the Mother Liquor. Grained massecuite only. Investigations upon the work of competent sugar boilers has shown that this test has a very important bearing upon economical boiling. The drop in purity may be very variable for equal purity of strike. Immediately after being drawn the sample is to be rapidly turbinated in the small laboratory centrifugal.

The author has devised special, light-weight, tin linings for the centrifugal; these can be kept on hand in any number, they slip in and out easily, collect the whole sample and do away with the necessity of cleaning the centrifugal. It is especially helpful in making a large number of such analyses.

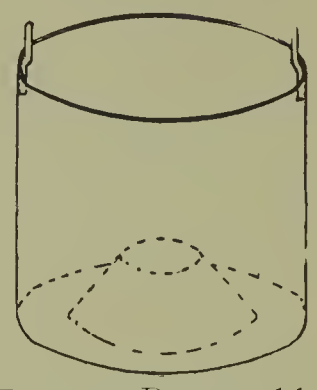


FIG. 5.—Removable Centrifugal Lining.

When the lowering of purity is abnormal, either way, the several causes to which it may be due should be investigated. The purity is determined after diluting to 18–20° Brix.

Polarization. This is made by taking either a normal or double-normal weight of the sample used for determining the Brix by double dilution, according to color, and proceeding as under the sugar, polarization, excepting that after filtration the solution must be acidulated before reading, viz.:

Fill a 50–55 cc. flask to the 50 cc. mark with the clear filtrate, add dilute acetic acid to faint acidity and complete to the 55 cc. mark with water; shake and polarize; increase the reading by one-tenth to compensate for the dilution.

When possible, clarify with neutral lead acetate and thus avoid later dilution.

EFFICIENCY OF CRYSTALLIZERS. This refers also to work carried on "at rest" in tanks or wagons. The mother liquor is separated by the laboratory centrifugal and its purity determined at 18–20° Brix, when the product is about half discharged from the vessel.

FINAL MOLASSES. This should invariably be weighed, being too viscid to measure and occluding much air.

Under a suitable arrangement one man can attend to the day's output. The well mixed day's composite sample is analyzed for, Brix by double dilution, sugar and purity (calculated).

Daily, a quantity in proportion to the amount made is set aside for the semi-monthly complete analysis, as detailed under sugar.

Control of the Boiling. Much can be accomplished at the pan-service tanks to bring about regulated, economical boiling. If they are uniform in size, deep, rather than broad, large rather than small and with facilities for rapid and thorough cleaning, much will have been gained. The several molasses are diluted to an exact Brix, generally 60° and heated to 70° C. at a "blow-up" station

over these tanks. Every tank and piece of apparatus in the factory should bear a number. These tanks should be graduated to 10 hectoliter units, vertically, upon a scale having an indicator actuated by a float.

Before the contents of a tank is started for feeding the vacuum pan the following data concerning the material are to be entered upon the sugar-boilers blackboard: Date, hour, tank number, name of material, temperature, Brix, and where possible, the purity.

Upon the basis of this data the massecuite purity is established and the finished product should agree within two points of the estimated.

Weights and Measures as Affecting Mill Extraction. (1) The *Sugar Cane* is bought by the Spanish ton as a matter of tradition. From the time it enters the factory the metric system should apply. It suffers variable shrinkage in weight before it is ground and proper chemical control has necessitated the present policy of weighing it upon a beam scale at the hoist just before discharging into the mill hopper.

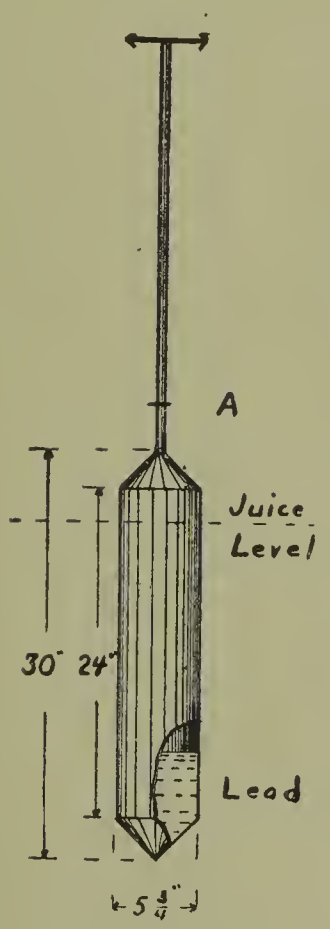


Fig. 6.—Float.

(2) *The Mill Raw Juice* is generally measured and when properly carried out, this will furnish a basis of calculation for the amount of sugar introduced (the real object) quite as satisfactory as weighing. Foam and occluded air have been the cause of some agitation in favor of weighing the juice, but this entails considerable expense that the author does not consider always justified. The installation of measuring tanks on the capacity basis of 3 hectoliters net per ton of cane per hour (i. e., 150 hectoliters, net for 1200 tons daily capacity) eliminates occluded air to a negligible quantity, while a float of special size and shape enables the true level of the juice to be determined with exactness and simplicity. This is shown in Fig. 6. It is made of galvanized iron, conical at both ends, weighted with 9½ lbs. of lead and provided at the top with a tube 2 inches long for holding a very light rod of wood, which in turn bears a double arrow and which is brought to a fixed point upon a scale.

The point "A" also serves as a definite spot from which to gauge the juice level for juices of different densities. The accompanying table shows the fluctuating juice level with varying Brix, the tank capacities being calculated accordingly. These levels are actually determined by floating the instrument in diluted molasses of juice density in a tall cylinder. The float rises and fails in a cage. It rests by far for the greater part well below that part of the juice containing the unliberated air and allows no air to collect below it. It measures to within one-sixteenth inch under working conditions.

Brix Reading.	Distance A, to Juice Level.
21.3	17.3 cm.
15.6	15.9 cm.
9.6	14.5 cm.
4.8	13.2 cm.

The specific gravity of the juice may be adjusted for varying temperature by the use of Gerlach's table.

Measuring tanks should be calibrated by weighing water into them until duplicate weighings agree within the polariscopic error as determined by the limits of the volumetric method using the Schmidt table.

(3) *Water of Saturation.* The average temperature of this must be known in order to arrive at its weight, as it is probably always measured. Duplicate tanks serve the purpose of measuring, well, but a good water meter is sufficient, provided simple means are at hand for occasionally checking it, under actual working conditions.

(4) *The Bagasse.* The weight of this, as determined by the formula, Cane + Saturation Water — Raw Mill Juice = Bagasse, is fully satisfactory, since the chemical control fails, in any case, where any one of the quantities is in error.

Stock Taking. The short season requires frequent stock taking and the author recommends that this be taken once a week until it has been demonstrated that the factory is working normally, but after this, with cane work, once a month is sufficient for the fully detailed report. For this weekly check, advantage should be taken of a stop and all the products of the factory composited into one laboratory sample upon the basis of the several volumes, when one analysis and one calculation will give the desired information.

THE SUGAR ACCOUNT.

All the sugar in the cane must be accounted for and brought up to a sum total of 100 per cent. The following form is a good general example.

General Sugar Account.	Per Cent, Basis of Cane.	Sugar in Cane 100 Per Cent.	Per Cent, Basis of Juice.	Tons.
Sugar in first sugar	9.14	72.67	78.97	4070.5
" second sugar	1.11	8.84	9.59	494.5
" total sugar	10.25	81.51	88.56	4565.0
" press cake	0.06	0.53	0.58	29.6
" final molasses	1.16	9.22	10.02	516.5
" undetermined loss	0.10	0.77	0.84	43.3
" mill raw juice	11.57	92.03	100.00	5154.4
" bagasse	1.00	7.97	.....	446.5
" cane	12.57	100.00	.....	5600.9

In a full technical account the above represents about 20 per cent of the data, the rest including averages of the analyses made of all the products, together with tons of cane ground, bags of the various grades of sugar made, time lost for different causes, data connected with the mill efficiency and the percentage yield of commercial sugars upon the basis of the cane.

The undetermined loss is due chiefly to the impossibility of accounting for all material involved in any undertaking.

There is loss from spilling, from inversion, long action of heat and errors in weights and measures with limits in accuracy of analyses. When this figure is 1 per cent of the total sugar in the juice it indicates good work, when it is 0.5 per cent it is excellent work.

General Methods.

Brix by the Hydrometer. All solutions up to 70° Brix are to be tested by the hydrometer directly, after the removal of air bubbles.

If the reading is not made at either 17½° C. or 20° C. corrections will be made by means of tables to be found in any standard text-book.

Double Dilution Method. (For products of over 70: Brix.) —The absence of cold water in tropical work precludes the cooling of solutions that have once been heated. The following method has been found most practicable: Use nickel plated, copper beakers of such size that the fist may readily be introduced. Select two of about equal weight and place upon opposite pans of the balance; from the heavier, file or cut off the material around the upper edge until they exactly balance. With the beakers now upon opposite pans of the balance, in one place about 400 grams or any convenient quantity of the material; into the other pour water until exact balance is secured; remove the beakers from the balance and pour the water of the one into the other containing the material; by means of the hand, mix the two until the last grain is dissolved, do not remove the hand until the operation is complete, in fact, endeavor to keep the hand equally submerged all the time.

Allow to stand until air has risen, take the Brix by the hydrometer, correct to standard temperature and multiply the result by 2. If the beaker used for water be only lightly greased within, it will deliver the water to the other to the last drop, thus obviating pouring back.

The Dilution to 18 to 20 Brix.—For rapid control of the process of boiling, based upon the purity, all products will be reduced to uniform density within these limits before making the test. Simple as it seems, the average chemist is longer in learning to perform this test with unfailing accuracy than any other test in sugar manufacture, this being especially true with products containing grain in suspension. The fault lies in losing some of the material by spilling before all the grain has been dissolved or before the mixture is absolutely uniform.

(1) For products positively known to contain no grain.

Select a cylinder 15 inches tall by 1½ inches diameter, having the upper edge of such a shape that it may be perfectly sealed by the palm of the hand; fill about two-thirds with water, pour in from about 110–120 cc. of the material, adding more water until within about 1½ inch from the top; seal tightly with the palm of the hand and shake vigorously until mixture is intimate; the result should always be a solution too dense rather than too thin; pour out a portion and add water if trial test shows too dense, mixing as before; continue this until proper figure is reached.

(2) For products known or suspected to have grain. Select two-liter enamelled cups of unbroken surface—these have no corners in which the grain may lodge and thus escape solution. Pour into the cup about 500 cc. of water, add about 200 cc. of the material and by means of the hand manipulate the mass until no more grain can be felt; transfer the solution to the cylinder and proceed as under (1) until the density is reduced to 18–20° Brix.

Dry Substance. In the cane sugar industry this is a purely empirical process and close conformity to certain conditions are necessary to secure even comparative results. The breaking down of the levulose molecule at temperatures above 80° C., the oxidation of non-sugars, the formation of acids that in turn produce more invert sugar which continues to decompose, make it impossible to dry to constant weight at atmospheric pressure, therefore a vacuum should be employed, when the temperature is held at 70° C., the vacuum should not be under 25 inches and a slow current of dry air allowed to pass through; weigh every 2–3 hours until constant in weight.

In the absence of any vacuum the following method may be used. It is based upon the official method of the Association of Official Agricultural Chemists, *Bull.* 107 (revised), U. S. Bureau of Chemistry, p. 64.

Place in a light crystallizing dish, provided with a watch-glass cover and stirring rod, 25 grams of broken glass in quite uniform pieces the size of coarse sand and washed free from dust. Dry quickly at 120–140° C., cool in a desiccator and weigh. Of sugar, take 10 grams; of molasses, 5–6 grams; of mill raw juice, 35 grams. Dry for exactly 10 hours at 98–100° C. (boiling water jacket). The time for drying the juice should begin with the disappearance of the water.

Sulphated Ash. About 3 grams of sugar are used and a proportionate amount of other products; if moisture exceed 25 per cent, as in cane juice, the water must be evaporated on the water bath. The true ash is calculated by multiplying by factor 0.9.

Condensed Waters. These include all the main hot water collectors, the pan and evaporator tail-pipes. To be tested for sugar with alpha-naphthol. Some form of continuous sampler is advised.

The Test. (Fruhling and Schultz, *Anleitung,* p. 166.) To 2 cc. of the suspected liquid add 5 drops of a 20 per cent alcoholic, sugar free, alpha-naphthol solution, then 10 cc. of purest sulphuric acid and shake thoroughly. With 0.1 per cent sugar the color is so deep as not to be transparent; with 0.01 per cent a bright red wine.

Boiler Water. Determine the acidity of the boiler-feed water and the alkalinity of the boiler water every 12 hours.

For the test, take a small amount from each boiler in service, titrate with standard acid (1 cc. = 0.001 CaO), using methyl orange as indicator. Report as grams CaO per 100 cc. Report number of pounds soda-ash used per 24 hrs.

Preparation for Clerget Test and Invert Sugar. *Juices.* Determine the sp. gr. Clarify 500 cc., strained and air-free with neutral lead acetate sol. (50° Brix); dilute to 550 cc., shake well and filter. Make the direct reading at as near 20° C. as possible. To about 150 cc., add ignited Na_2CO_3 to scant phenolphthalein alkalinity, stir and let stand 15 minutes; filter. Use exactly 75 cc. for Clerget by the Herzfeld inversion method. Use about 5 cc. for invert sugar test.

Molasses. Wash 2½ normals into a 500 cc. flask, clarify with sol. lead-*b*-acetate, mix, rotate to expel air, dilute to the mark and filter; collect 250 cc., neutralize with glacial acetic acid, double the amount of acetic acid; throw the filter and precipitate into the clear liquor, mix thoroughly and again filter. Polarize and multiply by 2. Remove the lead with Na_2CO_3 and proceed as under Juices for Clerget Test and Invert Sugar, using 15–20 cc. for the latter. True sucrose should be calculated by the following formulae.

Juice

$$\frac{\dfrac{26 \times \text{invert pol.}}{68.18 \text{ sp. gr.}} + \text{direct pol.}}{142.66 - 0.5\ t}$$

Molasses

$$\frac{\text{Direct pol.} - \text{invert pol. (2.67)}}{142.66 - 0.5\ t}$$

Herzfeld's Inversion Method. This will be found in detail in any standard work. After inversion is complete and while still in the water bath, add 1 gram powdered zinc, heat for another

5 minutes, pass through cotton, wash the cotton until 100 cc. is obtained at 20° C.

Invert Sugar Determination. To exactly 10 cc. of Fehling's solution, blue and 10 cc. of Fehling's solution, white, in a 250 cc. flask, add the solution under test and enough distilled water to make 50 cc. Boil cautiously upon a square of asbestos, having a central hole, for 2 minutes, then cool quickly. Add 10 cc. of 20 per cent solution KI, rotate, add 10 cc. of 25 per cent H_2SO_4, then titrate with N/10 thiosulphate. Work rapidly throughout, running in by 3–4 drops at the end.

Run a "blanc" upon the Fehling solution, under precise conditions of analysis to determine its value in terms of thiosulphate. Use the table of Meissl[1] and Hiller to calculate results. Careful determination by several chemists have shown that not all of the copper present can be accounted for with cane products and the following table of factors has been worked out:

TABLE OF INVERT SUGAR FACTORS

For use where the copper reduced is determinined by difference

Cc. Decinormal Thiosulphate Used.	Juices.	Sugars.		Molasses.				
	$I = \pm 6$	$I = \pm 1.2$	$I = \pm 3.4$	$I = \pm 15$	$I = \pm 32$	$I = \pm 39$	$I = \pm 46$	$I = \pm 55$
5	0.981	0.928	0.950	0.926	0.996	0.934	0.960	0.956
10	0.998	0.919	0.966	0.961	1.013	0.980	0.983	0.988
15	1.007	1.013	0.989	1.011	1.017	1.011	0.988	1.010
15.5	.....	.....	.....	.....	.....	.....	.....	1.015
20	1.016	1.048	1.016	1.021	1.020	1.032	1.001	1.001
25	1.045	.....	1.030	0.963	1.022	1.047	1.002	0.971

Example. Used in "blanc" titration, 27.75 cc., in back titration, 6.93 cc., net utilized, 20.82 cc. $0.00636 \times 20.82 = 0.1324$ gram Cu, $\frac{0.1324}{2} = 0.0662 = Z$. $W = 8.275$ grams material taken for determination. Polarization = 10.08. $0.0662 \times 100/8.275 = Y = 0.8$ per cent, $100 \times 10.08/10.08 + 0.8 = 92.7 = R$. $100 - 92.7 = 7.3 = I$. Since 20 cc. of Fehling's solution were used

[1] "Spencer's Handbook for Cane Sugar Manufacturers," pp. 129, 130.

instead of 50 cc., Cu/2 or 0.0662 gram must be multiplied by 2.5 to find the factor, F., which equals 0.165 gram Cu. By the Meissl and Hiller table, factor = 53.1. Hence 0.1324 × 53.1/8.275 = 0.85 approximate invert. Referring to the above table of corrections results in that of 1.016. 0.85 per cent × 1.016 gives 0.864 per cent corrected.

Attached will be found a Comprehensive Table of Purities arranged by the author for laboratory use.

The Table includes all purities that will occur in natural products and factory products between Brixes 5 and 30 and Sugars 1 to 28.5 per cent. The Brixes ascend by 2/10ths, the Sugars by 1/10th. The Purities are arranged in four blocks; beginning with the bottom, first block for sugars 1 per cent to 5.4 per cent; second block, sugars 5.5 per cent to 13.1 per cent; third block, sugars 13.2 per cent to 20.8 per cent; fourth block, sugars 20.9 per cent to 28.5 per cent.

765 Westminster Road,
Brooklyn, N. Y.

TABLE OF PURITIES FOR USE IN CANE AND BEET SUGAR FACTORIES

ARRANGED BY DAVID L. DAVOLL, JR.

COPYRIGHTED, 1913, BY DAVID L. DAVOLL, JR.

EXPLANATION OF AND MODE OF USING TABLE OF PURITIES

This Table of Purities has been arranged especially for laboratory use by the author. It may be framed in the usual way and the lines followed with a common ferule, or better, be pasted upon a cylinder $5\frac{3}{4}$ inches in diameter made to revolve upon a vertical axis, one Brix column being cut off and pasted to a fixed edge set close to the cylinder, when, by simple rotation, any sugar column may be brought up close to the Brix column and the purity noted.

The Table includes all purities that will occur in natural products and factory products between Brixes 5 and 30 and Sugars 1 to 28.5%. The Brixes ascend by 2/10ths, the Sugars by 1/10th. The Purities are arranged in four blocks: beginning with the bottom, first block for sugars 1 to 5.4%; second block, sugars 5.5 to 13.1%; third block, sugars 13.2 to 20.8%; fourth block, sugars 20.9 to 28.5%.

If, for instance, the purity corresponding to Brix 23.8 and sugar 18.9% is desired, by noting the regularly ascending scale for the sugar per cent the figure 18.9 will be met with in the third block; by now following down the Brix column at the extreme left, 23.8 will be found; the point of intersection of column under 18.9% sugar and opposite degree Brix 23.8 gives the Purity as 79.4.

Special space has been devoted to purities required for dilutions to 18–20° Brix in vacuum pan and crystallizer control, the Brix figures being repeated so as to permit of purity extension to embrace all purities between 30 and 94.

THE ASPHALTIG ROCKS OF THE UNITED STATES AND THEIR USE IN STREET PAVING

By S. F. PECKHAM

The word rocks in this paper is used in its geological sense, and comprises all varieties of mineral aggregate that may be saturated with bitumen. These rocks are found for the most part west of the Mississippi River. There are deposits of limited extent in Kentucky and Alabama, but they have not been entered commercially to any extent.

I was once shown a specimen of bituminous limestone which was said to be obtained in a quarry in Michigan, but I have never been able to locate it. The deposits west of the Mississippi River, however, extend from Utah through Oklahoma and New Mexico into Texas. They have been worked in Utah and Texas but from an economical standpoint are almost wholly confined to Oklahoma, where according to a recent report of the State Geologist, Dr. Gould, they are of sufficient extent to furnish material for paving all the cities of the United States.

On a recent trip to the Southwest extending to the Pacific coast, I had unusual opportunities for learning all the facts that may be stated in relation to these deposits in Oklahoma and their uses.

Starting from New York on June 9th, I visited first Chicago, then going south, Memphis, and west to McAlester, Okla. I spent a month in the vicinity of that city including a side trip to Ardmore, Okla., where I spent the 4th of July, and a few days after leaving McAlester. I spent two days in Ft. Worth, Texas, continuing my journey to El Paso, thence to Los Angeles where I remained two weeks, thence north to Ventura where I remained three weeks, then a week at Nordhoff, thence continuing north to Santa Barbara up the coast to San Francisco.

Leaving San Francisco by the Union Pacific Railroad, I spent two days in Salt Lake City, two days in Denver, three days in

Kansas City, one day in Louisville, two days in Washington, thence returning home to New York.

In all of these cities I took particular pains to observe and inform myself concerning the condition of the streets, and the extent to which in each of the cities mentioned so called asphalt pavements had been used for paving purposes.

The condition of the streets in Chicago might be said to be good to indifferent. Michigan Avenue which is used largely by automobiles and which consequently receives a continuous sprinkling of lubricating oil which keeps the surface of the asphalt soft and pliable, appears to be in splendid condition, but side streets are more or less filled with holes and indicate that wrong material was used although the repairs expended upon them were first-class in execution.

In McAlester, Okla., the asphalt streets were all comparatively new, having been laid but a few years. They were soft and wavy in their surface, easily impressed with the shoes of horses, and in many instances showed rutting from the action of wheels, all of which indicate that the materials used were of indifferent value. I asked a citizen of the city why such streets had been laid when they had at their doors some of the most valuable materials for street paving in the world. He replied that the streets were the result of competitive bidding. The lowest bidder received the contract, upon which comment is unnecessary.

At Ardmore, Okla., I walked over some magnificent streets, some of which had been laid five or six years. In several instances the excessive cold weather of the previous winter had developed cracks which had not fully closed, but on the whole the streets of the town which were laid wholly of materials obtained in the neighborhood were in magnificent condition. I asked if any tests had been applied to those streets other than the wear of farm wagons. I was told that the corrugated wheels of traction engines some of which were very heavy made no impression on those streets, and that the material of Sell's Circus had been unloaded on that particular street without leaving a scratch behind it.

Proceeding to Ft. Worth, Texas, I was fortunate in receiving an invitation from a friend to take an automobile ride over some of the streets of the city. These streets included natural rock asphalt, so called artificial asphalt and bitulithic surfaces. There was no diffi-

culty in distinguishing these streets by the action of the automobile. The natural rock asphalt streets were as level as a house floor, the bitulithic streets were wavy in every surface to such an extent that in some instances the automobile wheels bounded from one wave to another. The artificial asphalt streets were in various conditions.

There were two streets that were laid a number of years ago of the bituminous shell limestone, that occurs at Cline, southwest of San Antonio, Texas. This shell limestone saturated with bitumen in its natural condition is exceedingly tough and broken with great difficulty. After being ground and heated it resolidifies, but as the shells are broken into small pieces it is only held together by the bitumen, which becomes quite brittle. The streets laid of this material had gone to pieces under the wear of heavy traffic and were in very bad condition.

One of the finest streets in Ft. Worth and indeed one of the finest I have ever seen is something over a mile in length laid three years, and is made of a mixture of bituminous sandstone and bituminous limestone obtained near Ardmore. It was free from defects of any kind and looks as though those who laid it had finished their job the day before.

No opportunity was given me to examine any streets in El Paso, as I stopped there only two hours in the night.

When I reached Los Angeles I was particularly interested to examine carefully the streets of that city, as I was informed that $4,000,000 had been expended on streets the previous year. The centre of the city is well paved, and almost exclusively with asphalt, the basis of which is obtained from distillation of the petroleum produced in the neighborhood. The city stands in a region where the ground never freezes, consequently the heaving due to the excessive frost appearing in northern climates has never to be reckoned with. The streets as a general thing present a fine appearance, but complaints were made by citizens that the surfaces had to be frequently renewed and were a source of enormous expense.

Outside the city there are several boulevards which have been constructed into oiled roads within recent years. They too were regarded as very expensive in consequence of incessant repairs, and while the great Sierra Madre Boulevard was a highway that it was a pleasure to drive over, the construction of some highways in less

densely populated sections had proven to be more or less failures on account of defects due to excessive or insufficient mixture of oil with soil.

In Ventura County where a few years ago the advent of an oiled road was hailed with acclamation they were abandoned as impracticable for the above reasons. This matter of oiled roads, however, is a side issue, as they are not strictly speaking asphalt streets.

In Santa Barbara there are several miles of very fine asphalt streets. In San Francisco the condition of the streets was very similar to that of Los Angeles. Presumably the streets in all the towns of the Pacific Coast are at present laid and repaired with mixtures made from the residuum of California petroleum. In all these Pacific Coast towns the newer streets have been laid with either stone or brick gutters. In some instances the stone was artificial, in others it was concrete, but whatever the material might be the gutter was a separate construction from the street, and received not only a maximum of wear from the action of the water, but also the drippings of standing automobiles and the stamping of horses tied to posts. The advantage of this arrangement was apparent to the most casual observer, for it is well known that in asphalt street construction gutters have been a serious problem for many years.

On reaching Salt Lake City the magnificent wide streets of that place immediately attract attention. Their main streets were constructed in such a manner that with stone gutters and a strip from four to six feet of Belgian block, the wear of the street from heavy traffic, from the stamping of horses, etc., was completely removed from the centre of the street which was laid with asphalt that received less than one-half of the ordinary wear to which such streets are subjected. The result was an almost uniform condition of the streets that was so superior to anything I had seen elsewhere, that I was ready to exclaim that Salt Lake City had the finest streets in the whole country. A closer inspection, however, showed that the material from which these asphalt streets were constructed was soft and not impervious to the influences of the weather. Nevertheless, Salt Lake City has many of the finest asphalt streets that I have ever seen.

In Denver the streets were narrower, and while they had

stone or brick gutters, there were no strips of Belgian block to receive the heaviest wear of traffic.

I had expected to obtain some interesting items of information when I reached Kansas City. This city has 200 miles of asphalt streets in every conceivable condition from almost completely bare concrete to the finest asphalt street that can be built. I rode over several streets that had been laid twelve or fifteen years before, with a mixture of Oklahoma natural bituminous rocks, that had never been repaired, the most remarkable demonstration of the value of this material for street paving that I had ever witnessed. One of these streets in particular, several blocks in length, looked as though it was not more than a year or two old.

In Louisville, although in the immediate neighborhood of the deposit of Kentucky bituminous rock, there were almost no asphalt streets, the main thoroughfares being laid with wood and presenting a beautiful appearance.

I rode over many miles of asphalt streets in Washington. They were in magnificent condition as I expected to find them. I observed that Washington was also laying the gutters of stone or brick. This is an innovation which is bound to bear good fruit.

Unless the practice in Washington has been changed from what it was under the administration of Mr. Dow, as is not probable, there are no streets in Washington which are not constructed of natural asphalt. By this term, natural asphalt, is not meant natural asphaltic rocks, but asphalt occurring as deposits of asphaltum in masses which are afterward mixed with sand and converted into a street surface. There have never been constructed in Washington to my knowledge, streets made from natural bituminous rock from any source. However, for many years, great care has been exercised in laying and repairing the asphalt streets of this city, the result of which care has been a general condition of the asphalt streets superior to that of any other city in the country.

Getting home to New York and Brooklyn, the wretched condition of our streets was more forcibly impressed upon my mind after what I had seen. Of course in central New York City, the streets are subjected to the incessant wear of heavy traffic, but there are residential streets in New York and Brooklyn, the condition of which requires the strongest apologies.

One block near where I reside has been laid possibly eight years,

but has been repaired every year for the last four years. The repairs of one season disappeared almost completely. Between the repairs of that year and the next succeeding one the bitumen disappeared completely, and the sand blew into the gutters leaving in many instances the bare concrete. With what material these repairs were constructed I am unable to say.

These various object lessons observed over such a wide area and under such varying conditions of climate and commercial considerations that affect the use of materials of different kinds, teach a very striking and impressive lesson. If streets can be laid, over which traction engines with their corrugated wheels fail to leave an impression, and if setting aside heavy traffic, residential streets can be laid with natural materials that require no repairs for fifteen years, and if the cheaper materials obtained in the distillation of petroleum, which are wholly unfit for streets sustaining heavy traffic do not last on residential streets more than three to five years without repairs, I ask why they should be used, with constant repairs and renewals when at a somewhat greater first cost materials which are available, in the long run must prove much more satisfactory and in reality cheaper in the end.

I have been acquainted with the development of this industry in all its details in Oklahoma for fifteen years. I was present in 1897 when Mr. C. O. Baxter, as agent of the Gilsonite Paving Co. of St. Louis, prepared the material in Oklahoma and laid those streets in Kansas City which have sustained an average amount of traffic for fifteen years without repairs. I have been informed that soon after these streets were laid, the Barber Paving Co., purchased the deposits of bituminous limestone in Oklahoma, and that since then no work has been done anywhere with the materials then used. It is a fact, however, that when the proposal was made to the City Government, in 1908, to use in Ardmore similar materials, a committee of citizens together with the City Engineer first experimented on the proper proportion of bituminous limestone which should be mixed with bituminous sand or sandstone to form the most satisfactory materials for paving the streets of that town. They succeeded after a few months of experimenting in laying a street which is almost faultless. The work was undertaken by a contractor who has enlarged his business and improved his methods until the streets which he has laid in Tulsa and Ardmore, Okla., and Ft. Worth,

Texas, are unequalled by any similar surfaces anywhere in the whole country. I was told that the severe cold of last winter gave them a trial which they had never before received, and that some of them contracted to such an extent that they cracked.

The party, who constructed these streets, has found that a softening material, that is really maltha, less fluid than petroleum, and softer than asphaltum, can be extracted by boiling water from the deposits of bituminous sand that exist in inexhaustible quantities twelve miles west of Ardmore.

Summit Avenue, in Ft. Worth, Texas, which represents his last and most successful achievement in street paving, was laid with a mixture of bituminous limestone or chalk mixed in proper proportions with bituminous sand, and the whole tempered with this natural maltha which he has obtained as above described. The result is an almost perfect street; perhaps it is safe to say that it is perfect, having been laid three years and no defect can be found in it, not even a contraction crack, and it is as level as a house floor without wave or buckling, and hard enough to resist rutting even when the temperature in the sun is above 100 degrees Fahr.

The streets in Kansas City that were laid in 1897 and have stood ordinary traffic for fifteen years were constructed of similar materials. It is not claimed that in competitive bidding these materials can be furnished at the same price for which a mixture of sand and petroleum residuum could be laid, but while the first cost is greater, the average cost for fifteen years is far less.

In the construction of a street the cost of grading is the same whatever may be the material used for a surface, also the cost of the concrete foundation. The difference in price relates only to the surface of the street which is in reality one of the least items in the total cost of construction.

If there are as Dr. Gould asserts, sufficient deposits of this material in Oklahoma to pave the whole country, why is such a problem left out of the calculations of those who have in charge the construction of streets in our large Eastern cities.

It is well known to those who are familiar with the technology of asphalt streets, that a certain allowance must always be made when criticism is indulged on any particular piece of work, for the skill which has been exercised in the mechanical performance of the case in hand; but, such variation in mechanical skill or the lack of

it, does not explain the difference in results which are evidenced in the examples set forth in the foregoing relation.

No amount of mechanical skill can convert bad materials into good streets, nor can a reasonable amount of the lack of it so far damage the result as to convert first-class materials into poor streets. This charitable view of the technology of asphalt paving cannot cover the multitude of sins laid at its door. There is a fundamental difference in the materials used in this technology and the sooner that fact is recognized the more rapidly will progress be made toward the realization of an ideally perfect street, whose good qualities shall be limited only by their inherent defects; that is to say, that a perfect asphalt street as such may not possess all of the good qualities of an ideally perfect street.

Placing a street made of European Neufchatel bituminous rock at the head of a list of such structures, the question to be solved would be, how far all the elements of perfection with which it is characterized can be realized in any structure made of similar materials, or in an imitation thereof.

The solution of this question is brought to the laboratory of a chemist for a determination of the identity of the materials as the primary consideration. This determination can be made nowhere else, for, if under given conditions two substances from different sources can be made into streets that are apparently identical in quality, that is no proof of identity in the materials used, nor is it a warrant that the apparent equality is not accidental.

In the laboratory proximate analysis supplemented by elementary analysis—no matter how difficult in execution these analyses may be—can alone determine the identity of the materials. In the years that have elapsed I have subjected these Oklahoma bitumens to hundreds of parallel tests with California asphaltum, and with petroleum residuum. They are not identical, nor are they identical with Trinidad Pitch or any other South American bitumen that has been brought to my notice.

These Oklahoma bitumens resemble the bitumens of Seyssel and Neufchatel more than any other bitumens with which I have compared them. The first and simplest observation is their permanence under natural exposures to the elements. California asphaltum decomposes under the atmosphere and disintegrates into a carbonaceous soil. Trinidad Pitch with less rapidity does the same thing.

In the latter case the result is a brown pulverulent substance that assumes prismatic forms. In the case of California asphaltum that has resulted from the decomposition of the outflows of maltha, the result of the decomposition is very similar to that of Trinidad Pitch, but, in the outcrops of veins of asphaltum that occur near Asphalto in Kern County, Calif., the decomposition proceeds from the surface downward resulting in a brown substance with rhomboidal fracture, resembling carbonate of iron. The outcrops of Oklahoma bituminous rocks on the surface, though covered with lichens and having the appearance of long exposure to the elements, exhibit immediately beneath the surface very little change, if any, from the conditions found in the interior of the deposit.

When the bitumen is extracted from all of these materials, and subjected to parallel tests of hardness and flexibility, at low temperatures, it is found that there is no parallelism between these natural bitumens and the so called artificial bitumens or petroleum residuums. While those from Oklahoma become more dense, they remain flexible or at least elastic at zero Fahr.; while the other bitumens, either natural or artificial become brittle and fragile. These facts will explain the different results obtained when these different materials are used for street surfaces. Another simple test, the result of which is very marked, consists in observing the effect of boiling water upon the bituminous rock. This test when applied to Neufchatel and Oklahoma rocks results in a complete separation of the bitumen from the mineral aggregate. On the contrary when this test is applied to bituminous rocks from other localities, the boiling water does not produce any separation.

In the years 1894, '95 and '97, during which I visited California, and the island of Trinidad, and spent nearly a year in Oklahoma, I gathered a complete set of specimens, several hundred in the aggregate, illustrating the points above stated. They embraced specimens from the Ojai ranch in California, where the decomposition of asphaltum assumed the appearance of reefs in a sterile soil that were covered with lichens; also a complete set illustrating the decomposition products found at Asphalto; also a large number of specimens from deposits of bituminous limestone and bituminous sand in the vicinity of Ardmore, Okla; also a complete set of specimens illustrating the different materials found at the Pitch Lake in Trinidad.

I hoped for a number of years to be able to make an exhaustive comparative research on these different forms of natural bitumens, as well as the artificial residuum from petroleum. I had barely commenced the work on Trinidad Pitch when the Civil Engineer, who controlled my work, ordered me to desist. I have published what little I had accomplished on Trinidad Pitch, but the bulk of the work remains undone. I have given the specimens to the Museum of the Brooklyn Institute. The work is a work of years and awaits the labors of a chemist competent to undertake it, who loves chemical research next to his own soul.

I wish in this connection to express my thanks to Mr. Nelson H. McCoy, Secretary of the Chamber of Commerce of Ardmore, Okla., who has for many years assisted me in numberless ways in my researches, also to Mr. Clark R. Mandigo, Assistant City Engineer of Kansas City, Mo., who extended to me in the most courteous manner every facility for learning the exact condition of the asphalt streets of Kansas City.

CODE OF ETHICS

Prepared in accordance with a vote of the Institute at the Washington Meeting and amended at New York Meeting by Committee on Professional Ethics.

G. W. THOMPSON, Chairman
CHAS. F. McKENNA, A. C. LANGMUIR, A. D. LITTLE

ARTICLE I.

PURPOSE OF THE CODE:

To define the rules of professional conduct and ethics for the members of the Institute.

ARTICLE II.

THE INSTITUTE EXPECTS OF ITS MEMBERS:

1st. That in all their relations, they shall be guided by the highest principles of honor.

2d. The upholding before the public at all times of the dignity of the chemical profession generally and the reputation of the Institute, protecting its members from misrepresentation.

3d. Personal helpfulness and fraternity between its members and toward the profession generally.

4th. The avoidance and discouragement of sensationalism, exaggeration and unwarranted statements. In making the first publication concerning inventions or other chemical advances, they should be made through chemical societies and technical publications.

5th. The refusal to undertake for compensation work which they believe will be unprofitable to clients without first advising said clients as to the improbability of successful results.

6th. The upholding of the principle that unreasonably low charges for professional work tend toward inferior and unreliable

work, especially if such charges are set at a low figure for advertising purposes.

7th. The refusal to lend their names to any questionable enterprise.

8th. Conservatism in all estimates, reports, testimony, etc., especially in connection with the promotion of business enterprises.

9th. That they shall not engage in any occupation which is obviously contrary to law or public welfare.

10th. When a chemical engineer undertakes for others work in connection with which he may make improvements, inventions, plans, designs or other records, he shall preferably enter into a written agreement regarding their ownership. In a case where an agreement is not made or does not cover a point at issue, the following rules shall apply:

a—If a chemical engineer uses information which is not common knowledge or public property, but which he obtains from a client or employer, any results in the form of plans, designs or other records shall not be regarded as his property, but the property of his client or employer.

b—If a chemical engineer uses only his own knowledge or information or data, which by prior publication or otherwise are public property, and obtains no chemical engineering data from a client or employer except performance specifications or routine information, then the results in the form of inventions, plans, designs or other records should be regarded as the property of the engineer and the client or employer should be entitled to their use only in the case for which the engineer was retained.

c—All work and results accomplished by the chemical engineer in the form of inventions, plans, designs or other records, or outside of the field for which a client or employer has retained him, should be regarded as the chemical engineer's property.

d—When a chemical engineer participates in the building of apparatus from designs supplied him by a client, the designs remain the property of the client and should not be duplicated by the engineer nor anyone representing him for others without express permission.

e—Chemical engineering data or information which a chemical engineer obtains from his client or employer or which he

creates as a result of such information must be considered confidential by the engineer; and while he is justified in using such data or information in his own practice as forming part of his professional experience, its publication without express permission is improper.

f—Designs, data, records and notes made by an employee and referring to his employer's work, should be regarded as his employer's property.

g—A client does not acquire any exclusive right to plans or apparatus made or constructed by a consulting chemical engineer except for the specific case for which they were made.

11th. A chemical engineer cannot honorably accept compensation, financial or otherwise, from more than one interested party, without the consent of all parties; and whether consulting, designing, installing or operating, must not accept compensation directly or indirectly from parties dealing with his client or employer.

When called upon to decide on the use of inventions, apparatus, processes, etc., in which he has a financial interest, he should make his status in the matter clearly understood before engagement.

12th. The chemical engineer should endeavor at all times to give credit for work to those who, so far as his knowledge goes, are the real authors of such work.

13th. Undignified, sensational or misleading advertising is not permitted.

14th. Contracts made by chemical engineers should be subject to the Code of Ethics unless otherwise specified.

ARTICLE III.

For the administration of this Code of Ethics, a Committee on Ethics shall be appointed by the president holding office at the time of the adoption of this Code with the approval of the Council, to consist of five members; one appointed for five years, another for four years, another for three years, another for two years, another for one year, and thereafter, the president then holding office shall appoint one member annually to serve for five years and also fill such vacancies as may occur for an unexpired term. All of these members shall be over forty years of age. The Committee shall elect its own chairman. The Committee on Ethics shall investigate all complaints submitted to them bearing upon the professional con-

duct of any member, and after a fair opportunity to be heard has been given to the member involved, shall report its findings to the Council, whose action shall be final.

ARTICLE IV.

Amendments.

Additions to or modifications of this Code may be made according to Article VIII of the Constitution.

CONSTITUTION

ARTICLE I.

NAME.

This organization shall be termed,

AMERICAN INSTITUTE OF CHEMICAL ENGINEERS

ARTICLE II.

OBJECTS.

The objects of this organization shall be:

To advance the cause of applied chemical science.

To give the profession of Chemical Engineers such standing before the community as will justify its recognition by Municipal, State, and National authorities in public works.

To raise the professional standard among Chemical Engineers, discouraging and prohibiting unprofessional conduct.

To coöperate with educational institutions for the improvement of the education of the men who are to enter this profession.

To encourage original work in chemical technology.

To promote pleasant acquaintance and social and professional intercourse among its members.

To publish and distribute such papers as shall add to classified knowledge in chemical engineering and shall increase industrial activity.

ARTICLE III

MEMBERSHIP

SECTION 1. (*Qualifications for Membership.*) Membership shall consist of two grades: Active and Junior.

ACTIVE MEMBERSHIP shall require the following preparation and training:

All candidates must be not less than 30 years of age and must be proficient in chemistry and in some branch of engineering as applied to chemical problems, and must at the time of election be engaged actively in work involving the application of chemical principles to the arts. All candidates for admission to this Institute are expected to have *expert knowledge of at least one branch of applied chemistry.* and must fulfill one of the following requirements:

1. Candidates who hold no degree from an approved university or technical school must have had ten years' experience in chemical technology; five being in responsible charge of operations requiring the elaboration of raw materials, the design of machinery involving chemical processes, or the application of chemistry to industry.

2. Candidates who hold the degree of A. B. (Bachelor of Arts) from an approved university or technical school offering a four-year course must have had at least eight years of practical experience as outlined under No. 1.

3. Candidates who hold the degree of Ch. E. (Chemical Engineer), B. S. (Bachelor of Science), in Chemistry or Chemical Engineering, or E. E. (Electrical Engineer), C. E. (Civil Engineer), or M. E. (Mechanical Engineer), or equivalent degrees from an approved university or technical school offering at least a four-year course, must have had at least five years' practical experience as outlined under No. 1.

4. For candidates who in addition hold the degree of Ph. D. (Doctor of Philosophy) or Sc. D. (Doctor of Science) in Chemistry, the number of years required to earn the higher degree may be deducted from the number of years of experience required.

Junior Membership shall require the following preparation and training:

All candidates must be not less than 23 years of age and must be engaged, at the time of election, in some branch of applied chemistry and must fulfill one of the following requirements:

1. Hold the degree of Ch.E. (Chemical Engineer), B.S. (Bachelor of Science) in Chemistry or Chemical Engineering, E.E. (Electrical Engineer), C.E. (Civil Engineer), M.E. (Mechanical Engineer), or equivalent degree from an approved university or technical school offering at least a four years' course.

2. Have had five years' experience in Applied Chemistry.

Junior Members shall have all privileges of the Institute excepting those of voting, holding office, and wearing the emblem or badge of Active Membership. A suitable emblem or badge of Junior Membership as adopted by the Institute may be worn by the Junior Members. When qualified, a Junior Member may apply for Active Membership, but must do so before reaching the age of 35, otherwise his membership shall expire.

Section 2. (*Applications.*) All applications for membership must be made to the Secretary in writing, and shall embody a concise

statement with the dates of the candidate's professional training and experience, and shall be in a form and in such detail as may be prescribed by the Membership Committee. The applicant for Active Membership shall give the names of at least five members to whom he is personally known. The applicant for Junior Membership shall give the names of at least five persons to whom he is personally known, two of whom shall preferably be members of the Institute. Each of these shall be requested by the Secretary to certify to the training, experience, professional attainment, and standing of the applicant. On receiving a favorable report from at least three of these references, the applicant shall be eligible to recommendation by the Membership Committee.

Section 3. (*Election of Members.*) At stated periods the Secretary shall mail to the members a ballot containing a list of all applicants who have been recommended by the Membership Committee. This list shall contain a detailed statement of each applicant's career and the names of the members who have vouched for him. All ballots shall be returned to the Secretary not later than three weeks after the date of issue. The ballots shall be canvassed by the Membership Committee, who shall report to the Council, who shall then declare each applicant elected for whom at least ninety-five per cent. of all ballots cast are in the affirmative. Provided, however, that any member voting in the negative may address a confidential letter to the Council, stating his objections to the candidate with evidence for the charges made. If the Council upon investigation considers such objections valid, they may declare an election void. A rejected candidate may make application again any time after one year. Persons elected to membership shall be notified at once by the Secretary. They must then subscribe to the rules of the Institute.

Section 4. (*Honorary Members.*) As the result of unusual ability and public recognition on the part of the industrial world, a person may, upon nomination of the Council and a vote of the Society at large, be made an Honorary Member, but at no time shall this number exceed five.

Section 5. (*Expulsions.*) For abuse or misuse of the privileges of the Institute or conduct unbecoming a member in the opinion of the Council, a two-thirds vote of the Council may expel any member of the Institute.

Section 6. (*Dues.*) The entrance fee for Active Members shall be $15.00; Junior Members shall pay no entrance fee; Annual dues

for active members $15.00, for Junior Members $10.00. Junior Members, on becoming Active Members, shall pay an entrance fee o' $15.00 less $1.00 per year for each year of their membership as Junior Members. Provided, however, that no entrance fee shall be exacted until the membership shall reach 200.

Any member may anticipate his dues for life by paying in advance such a sum as would be demanded by any reputable insurance association to yield an annuity equal to the annual dues from the time of the agreement until death. Upon resignation, or expulsion, all money so provided is to become the property of the Institute. Any person joining the Institute after the middle of the fiscal year is required to pay one-half of the dues only for that year. Any person in arrears for three months shall be notified by the Secretary. For non-payment at the expiration of one-half year, a member forfeits the right to vote or to receive the notices of the Association until dues are paid in full. All members are considered as such unless actual resignations are formally presented and accepted with the full payment of dues. On account of extenuating circumstances, dues may be remitted to any member by a two-thirds vote of the Council.

ARTICLE IV.

OFFICERS.

Section 1. The officers of this Society shall be a President, three Vice-Presidents, a Secretary, a Treasurer, an Auditor, and nine Directors. The officers shall be elected at the annual meeting. The President shall serve one year, the Vice-Presidents for three years each, and the Directors for three years each. The Secretary, Treasurer, and Auditor shall be elected for terms of one year each. At the first annual meeting one Vice-President shall be chosen for one year, one for two years, and one for three years. Three Directors shall be chosen for one year, three for two years, and three for three years. Thereafter, officers shall be chosen annually to serve full terms. The President, Ex-Presidents for the two years succeeding the expiration of their term of office as President, Vice-Presidents, Secretary, Treasurer, and Directors shall constitute the Council of the Institute. The President, Vice-Presidents, and Directors cannot be re-elected within the current twelve months from the expiration of term. The duties of office begin immediately after election and notification. An acceptance of office must be in writing addressed to the Secretary. Vacancies occurring in any office shall be filled by a majority vote of the Council for the

unexpired term. The duties of all officers shall be such as usually pertain to their offices or may be delegated to them by the Council or the Institute.

Section 2. (*Election of Officers.*) After the election at which this Constitution is adopted, the election of officers shall be by letter ballot. The Secretary, at least eight (8) weeks prior to each annual meeting, shall send to every member of the Institute a blank nominating ballot upon which the member may make nominations for the officers and Directors to be elected at the coming annual meeting. The nominating ballot is then to be properly signed and transmitted to the Secretary not later than five (5) weeks prior to the annual meeting. It shall then become the duty of the Secretary to prepare and issue an official ballot upon which shall appear the names of all nominations for office or for Directors which shall have appeared upon at least ten (10) nominating ballots. The official ballots shall be mailed not later than three (3) weeks prior to the annual meeting, one to each member, who shall properly signify on it his choice for the various offices and Directors, and transmit it to the Secretary. At the annual meeting the President shall appoint tellers to whom the Secretary shall deliver all the ballots received by him unopened, and who shall count and announce the vote.

ARTICLE V

COUNCIL

The Council shall have supervision and care of all property of the organization, and shall conduct its affairs according to the Constitution and By-Laws. At each annual meeting it shall present a statement of its proceedings during the year. Eight members of the Council called together by notice from the Secretary shall constitute a quorum, provided, however, that three members may be represented by proxy.

ARTICLE VI.

STANDING COMMITTEES.

The Council shall appoint the following committees:

1. FINANCE.
2. COMMITTEE ON MEETINGS.
3. PUBLICATIONS.
4. MEMBERSHIP.
5. LIBRARY.
6 HOUSE COMMITTEE.

FINANCE COMMITTEE.

The Finance Committee shall have charge of the financial affairs of the Institute. This committee must prepare the budget and approve all expenditures. The Chairman of the Committee may be the Auditor of the Institute.

MEMBERSHIP COMMITTEE.

The Membership Committee shall be constituted of fifteen members, ten of whom may vote by proxy at any meeting. To the Membership Committee all applications for membership shall be referred. It is the duty of this committee to see that no person is admitted to the organization who is not qualified.

COMMITTEE ON MEETINGS.

This committee shall have charge of all meetings of the organization and shall fix dates and places of meeting.

COMMITTEE ON PUBLICATIONS.

This committee shall look after the papers presented to the Institute. If considered expedient, any or all of these papers may be published and distributed to members.

LIBRARY COMMITTEE.

This committee shall have charge of all permanent records, books, papers, pamphlets, etc., and shall obtain and place on file a complete record of all patent literature in reference to chemical engineering.

HOUSE COMMITTEE.

This committee shall look after the social affairs of the Institute, fixing the time and place of entertainments.

ARTICLE VII.

MEETINGS.

The annual meeting of the Association shall be held in December, the exact date to be fixed by the Council.

This Institute shall be governed by its Constitution in conformity with the laws of the United States. All questions shall be decided by majority of votes cast. The Institute shall not be held responsible for opinions expressed in papers. The name or use of the Institute shall not be tolerated for any commercial purpose.

Upon the adoption of this Constitution officers shall be elected immediately to hold office until the election and installation of their successors.

ARTICLE VIII.

AMENDMENTS TO THE CONSTITUTION.

Any member may propose an amendment by addressing the Secretary. At the first regular meeting thereafter the subject shall be discussed, and if worthy, notice to vote on same shall be posted until the next regular meeting, and written copy of the notice shall be sent to each member. The proposed amendment shall then be discussed in open meeting and can be passed by two-thirds vote of all members of the Institute as the result of letter ballot.

BY-LAWS

ORDER OF BUSINESS.

REGULAR MEETING.

Reading of minutes of last stated meeting.
Miscellaneous announcements.
Reading of papers, discussion, and communications.
Adjournment.

ANNUAL MEETING.

Reading of minutes of last stated meeting.
Miscellaneous announcements.
Stated business.
Annual reports.
Election of officers.
Address of retiring President, etc.
Adjournment.

In all questions requiring parliamentary ruling not provided for by the Rules of the Institute, "Robert's Rules of Order" shall be the governing authority.

OFFICERS AND COMMITTEES FOR 1913

COUNCIL

Elected at Detroit Meeting, December 7, 1912

President,

T. B. Wagner ..... New York, N. Y.

Vice-Presidents,

M. C. Whitaker ..... New York, N. Y.
R. K. Meade ..... Baltimore, Md.
G. W. Thompson ..... Brooklyn, N. Y.

Secretary,

John C. Olsen ..... Brooklyn, N. Y.

Treasurer,

F. W. Frerichs ..... St. Louis, Mo.

Auditor,

Geo. D. Rosengarten ..... Philadelphia, Pa.

Ex-Presidents,

F. W. Frerichs ..... St. Louis, Mo.
L. H. Baekeland ..... Yonkers, N. Y.

Directors for One Year

Edw. G. Acheson ..... Niagara Falls, N. Y.
Wm. M. Booth ..... Syracuse, N. Y.
Edw. Hart ..... Easton, Pa.

Directors for Two Years

A. C. Langmuir ..... Brooklyn, N. Y.
H. S. Miner ..... Gloucester City, N. J.
A. Bement ..... Chicago, Ill.

Directors for Three Years

Geo. D. Rosengarten ..... Philadelphia, Pa.
Jokichi Takamine ..... New York, N. Y.
Jas. R. Withrow ..... Columbus, Ohio

COMMITTEE ON PUBLICATIONS

FRERICHS, F. W., *Chairman*
ANDREWS, LAUNCELOT W.
HART, EDWARD
BAIN, J. WATSON
KIPPENBERG, HENRY
OLSEN, J. C.
ITTNER, M. H.

MEMBERSHIP COMMITTEE

LANGMUIR, A. C., *Chairman*
ADAMSON, GEO. P.
BASSETT, WM. H.
CONVERSE, W. A.
DECEW, J. A.
DOW, A. W.
ITTNER, M. H.
ROSENGARTEN, GEO. D.
KAUFMANN, H. M.
MINER, H. S.
OLNEY, L. A.
RICHARDS, J. W.
ROBERTSON, A.
THOMPSON, G. W.

CHAIRMEN OF LOCAL COMMITTEES ON MEMBERSHIP

FRERICHS, F. W.	St. Louis, Mo.
BELDEN, A. W.	Pittsburgh, Pa.
LIHME, I. P.	Cleveland, Ohio
ROSENGARTEN, GEO. D	Philadelphia, Pa.
BYERS, H. G.	Seattle, Wash.
PARKER, T. J.	New York, N. Y.
LITTLE, A. D	Boston, Mass.
CONVERSE, W. A	Chicago, Ill.

COMMITTEE ON CHEMICAL ENGINEERING EDUCATION

WITHROW, JAS. R., *Chairman*
BOOTH, WM. M.
SADTLER, SAMUEL P.
WHITAKER, M. C.
WIECHMANN, F. G.

COMMITTEE ON MEETINGS

SADTLER, SAMUEL P., *Chairman*
BOOTH, WM. M.
MINOR, JOHN C., Jr.
HOWARD, HENRY
LANGMUIR, A. C.
MEADE, R. K.
OLSEN, J. C.
SADTLER, S. S.

COMMITTEE ON BOSTON MEETING

Howard, Henry, *Chairman*
Chas. A. Catlin
Little, A. D.
Olney, L. A.
Sharples, S. P.
Thorp, F. H., *Secretary*

FINANCE COMMITTEE

Thompson, G. W., *Chairman*
Toch, Maximilian
McKenna, Chas. F.
Elliott, A. H.

COMMITTEE ON MEDAL

Booth, Wm. M., *Chairman*
Richards, J. W.
Robertson, A.
Sadtler, Samuel P.

COMMITTEE ON STANDARDIZATION OF BOILER TESTS

Bement, A.
Booth, Wm. M.
Campbell, J. H.
Prentiss, George N.

LIBRARY COMMITTEE

Alexander, Jerome, *Chairman*
Olsen, J. C.
Myers, Ralph E.

COMMITTEE ON PATENTS

Baekeland, L. H., *Chairman*
Grosvenor, Wm. M.
Toch, Maximilian
Whitaker, M. C.

COMMITTEE ON PUBLIC POLICY

McKenna, Chas. F., *Chairman*
Frerichs, F. W.
Baekeland, L. H.
Taylor, Edw. R.
Parker, Thos. J.
Takamine, Jokichi

COMMITTEE ON ETHICS

Thompson, G. W., *Chairman*, 5 yrs.
McKenna, Chas. F., 3 yrs.
Little, A. D., 2 yrs.
Langmuir, A. C., 1 yr.

LIST OF MEMBERS, JUNE, 1913

Honorary Member

CHANDLER, CHAS. F., Columbia University, New York City.

Active Members

ACHESON, EDWARD G., Niagara Falls, N. Y.
President, International Acheson Graphite Co.

ADAMSON, GEORGE P., 233 Reeder St., Easton, Pa.
Vice-President and General Manager, The Baker and Adamson Chemical Co.

ADGATE, MATTHEW, Naugatuck, Conn.
Supt. of the Naugatuck Chemical Co.

ALEXANDER, D. B. W., 1000 Date St., Los Angeles, Cal.
Pacific Coast Chemist for The Barber Asphalt Paving Co.

ALEXANDER, JEROME, 502 West 45th St., New York City.
Treasurer and Chemist, National Gum and Mica Co., National Glue and Gelatin Works.

ALLEN, LUCIUS E., Box 22, Belleville, Ont., Can.
Consulting Chemical Engineer, Managing Director Ontario Limestone and Clay Co., Ltd., Belleville, Ont.

ANDERSON, LOUIS J., 315 Burke St., Easton, Pa.
Chemical Engineer, Alpha Portland Cement Co., Easton, Pa.

ANDREWS, LAUNCELOT W., Davenport, Ia.
President, Andrews Chemical Works.

ARNOLD, CHARLES E., 602 West 20th St., Wilmington, Del.

AUSTIN, HERBERT, 485 North Main St., Fall River, Mass.
Chemical Engineer and Partner Manager of Ernest Scott & Co., of Fall River, Mass., and Montreal, P. Q.

AYER, ARTHUR W., 3403 Gray's Ferry Rd., Philadelphia, Pa.
General Supt., Harrison Bros. & Co.

BAEKELAND, LEO H., Yonkers, N. Y.
Research Chemist and Chemical Engineer.

BAIN, J. WATSON, University of Toronto, Toronto, Can.
Associate Professor of Applied Chemistry.

BAIRD, WM. H., 1199 Woodward Ave., Detroit, Mich.
Secretary, Larrowe Construction Co.

BAKER, JOHN T., Phillipsburg, N. J.
President, J. T. Baker Chemical Co.

BARTON, G. E., 227 Pine St., Millville, N. J.
In charge of Laboratory and Dept. Mfg. Glass, Whitall Tatum Co.

BARUCH, EDGAR, 806 Wright & Callender Bldg., Los Angeles, Cal.
Consulting Chemical Engineer.

BARTOW, EDWARD, Urbana, Ill.
Professor of Analytical Chemistry, Univ. of Ill. Director of State Water Survey of Illinois. Consulting Chemist with the Davenport Water Co.

BASSETT, WILLIAM H., Cheshire, Conn.
Metallurgist, American Brass Co.

BEBIE, J., 1800 South 2d St., St. Louis, Mo.
Chemical Engineer, Monsanto Chemical Works.

BECK, ARTHUR G., care Canada Cement Co.
Exchaw, Alberta, Canada.

BECNEL, LEZIN A., 51 Arabella St., New Orleans, La., P. O. Box 390.
Chemical Engineer and Consulting Chemist.

BEERS, FRANK T., Washburn, Wis.
Supt. Barksdale Plant, E. I. du Pont de Nemours Powder Co.

BEHREND, OTTO F., Erie, Pa.
Vice-President and Treasurer, Hammermill Paper Co.

BELDEN, A. W., Bureau of Mines, 40th & Butler Sts., Pittsburgh, Pa.
Engineer in Charge.

BEMENT, A., 206 S. LaSalle St., Chicago, Ill.
Consulting Mining and Mechanical Engineer.

BOOTH, L. M., 136 Liberty St., N. Y.
President and Director, L. M. Booth Co., New York.

BOOTH, WILLIAM M., Dillaye Building, Syracuse, N. Y.
299 Broadway, New York, Consulting Chemist and Engineer.

BOWER, WILLIAM H., 2815 Gray's Ferry Rd., Philadelphia, Pa.
First Vice-President of Henry Bower Chemical Mfg. Co.

BROOKS, PERCIVAL C., General Chemical Co., Chicago Heights, Ill.
Asst. Supt., Illinois Works, General Chemical Co., Chicago Heights, Illinois.

BROWN, H. F., Room 915, du Pont Bldg., Wilmington, Del.
Chemical Director, Smokeless Powder Dept., E. I. du Pont de Nemours Powder Co.

BRAGG, E. B., Evanston, Ill.
Vice-President and Manager of General Chemical Co., Chicago Branch.

BYERS, HORACE G., Seattle, Wash.
Professor Chemistry, University of Washington, Consulting Chemical Engineer.

CAMP, J. M., Chief Bureau of Instruction, Carnegie Steel Co., Carnegie Steel Co., Carnegie Bldg., Pittsburgh, Pa.

CAMPBELL, JOHN HAYES, 161 N. Catherine Ave., La Grange, Ill.
Chemical and Metallurgical Engineer, 2200 Insurance Ex., Chicago, Ill.

CATLIN, CHARLES A., 133 Hope St., Providence, R. I.
Chief Chemist and a Director of the Rumford Chemical Works.

CHUTE, HARRY O., 197 Pearl St., New York. Chemical Engineer.

CONVERSE, WILLIAM A., 2005 McCormick Building, Chicago. Ill.
Chemical Director Dearborn Drug & Chemical Works.

CONNER, ARTHUR B., 217 W. Boulevard, Detroit, Mich.
Chief Chemist and Chemical Engineer for Detroit Chemical Works, Detroit, Mich.

CORSE, WM. M., Sycamore St., & N. Y. C. Belt Line, Buffalo, N. Y.
Works Manager, Lumen Bearing Co., Buffalo, N. Y.

CROWLEY, CHAS. F., Omaha, Neb.
Gas Commissioner of the City of Omaha, Neb.; Professor of Chemistry, Creighton Medical College.

CUSHMAN, ALLERTON S., 19th and B Sts., N. W., Washington, D. C.
Director and President, Institute of Industrial Research.

DAILEY, J. G., c/o Clinchfield Fuel Co., Spartanburg, S. C.
Chemical Engineer, Clinchfield Fuel Co., Spartanburg, S. C.

DANNENBAUM, HERMAN, Frankford, Philadelphia, Pa.
Vice-President National Ammonia Co.

DAVOLL, DAVID L., Jr., 765 Westminster Road, Brooklyn, N. Y.
Chief Chemist, Henry Heide, 313 Hudson St., New York, N. Y.

DEAN, JOHN G., Box 610, Los Angeles, Cal.

DECEW, J. A., Canadian Express Bldg., Montreal, Canada.
Consulting Chemical Engineer.

DILLER, H. E., c/o General Electric Co., Erie, Pa.
Chemist and Metallurgist, Research Laboratory, General Electric Co., Erie, Pa.

DOW, A. W., 131 E. 23d St., New York, N. Y.
Member of the firm of Dow & Smith, Consulting Engineers.

ELLIOTT, A. H., 52 E. 41st St., New York, N. Y.
Consulting Engineer.

ELLIS, CARLETON, 92 Greenwood Ave., Montclair, N. J.
Consulting Chemist and Inventor.

FOERSTERLING, HANS, 380 High St., Perth Amboy, N. J.
Second Vice-President, Roessler & Hasslacher Chemical Co.

FOWLER, THEODORE V., Box 15, Buffalo, N. Y.
Supt. of the Buffalo Works of the General Chemical Co.

FRASCH, HERMAN, 17 Battery Place, New York, N. Y.
President Union Sulphur Co., 17 Battery Place, New York, N. Y.

FRENCH, EDW. H., Smethport, Pa.
Chemical Engineer, Hilton & French, Smethport Research and Testing Laboratories.

FRERICHS, F. W., 4320 Washington Bou., St. Louis, Mo.
Vice-President, Herf & Frerichs Chemical Co.

GIBBS, A. E., c/o Pennsylvania Salt Mfg. Co., Greenwich Point, Phila., Pa.

GLOVER, H. LESTER, 73 W. Johnson St., Germantown, Phila., Pa.
Supt. of the Falls of Schuylkill Works of the Powers-Weightman-Rosengarten Co.

GRAY, CHAS. W., Driftwood, Pa.
Consulting Chemist, Keystone National Powder Co.

GRETH, J. C. WM., Pittsburgh, Pa.
Manager, Water Purifying Dept. of William B. Scaife & Sons Co.

GRISWOLD, THOMAS, Jr., Midland, Mich.
Engineer, The Dow Chemical Co.
Secretary, The Midland Chemical Co.

GROSVENOR, WM. M., 50 E. 41st St., New York City.
Consulting Chemist and Factory Engineer.

GUDEMAN, EDWARD, 903–4 Postal Telegraph Bld., Chicago, Ill.
Consulting Chemist and Chemical Engineer.

HAANEL, EUGENE, Dept. of Mines, Ottawa, Ont., Can.
Director of Mines, Dept. of Mines, Ottawa, Ont., Can.

HARRIMAN, NORMAN F., Union Pacific Laboratory, Omaha, Neb.
Chemist and Engineer of Tests, Union Pacific R. R. Co.

HART, EDWARD, Easton, Pa.
Prof. Chemistry, Lafayette College; President Baker & Adamson Co.; Prop. Chem. Pub. Co.; Consulting Engineer.

HEBDEN, JOHN C., Box 465, Providence, R. I.
Vice-President and General Manager, Franklin Process Co.

HERRESHOFF, J. B. FRANCIS, 620 West End Ave., New York, N. Y.
Vice-President Nichols Copper Co., Consulting Engineer General Chemical Co.

HOLLAND, WM. R., Gloucester City, N. J.
Foreman of the Chemical Dept., Welsbach Light Co., and Assistant to Chief Chemist.

HOLLANDER, CHARLES S., Hartford, Conn.
Secretary and Chief Chemist, Eastern Chemical Works, Inc., Hartford, Conn.

HOSKINS, WM., 111 W. Monroe St., Chicago, Ill.
Mariner & Hoskins, Consulting Chemical Engineers.

HOWARD, HENRY, 33 Broad St., Boston, Mass.
Vice-President, Merrimac Chemical Co.

HUGHES, L. S., 1246 Dearborn Ave., Chicago, Ill.
Chemist Illinois Steel Co.

HUMPHREY, H. C., 17 Battery Pl., New York City.
Chief Chemist, Eastern Branch, Corn Products Refining Co.

ITTNER, MARTIN H., Colgate & Co., Jersey City, N. J.
Chief Chemist, Colgate & Co.

JAMES, JOSEPH H., Pittsburgh, Pa.
Prof. Chemical Engineering Practice, Carnegie Technical Schools.

JONES, A. B., 981 Central Ave., Plainfield, N. J.
Supt. Laurel Hill and Bayonne Works, General Chemical Co.

JONES, L. C., Syracuse, N. Y.
Laboratory Manager, Solvay Process Co., and Semet Solvay Co.; Vice-President, Solvay Colleries Co.

KAUFMANN, H. M., 55 John St., New York, N. Y.
General Manager, Mutual Chemical Co. of America.

KILMER, FREDERICK BARNETT, 147 College Ave., New Brunswick, N. J.
Director of Laboratories, Johnson & Johnson, New Brunswick, N. J.

KIMMEL, H. R., 517–519 Superior Bldgs., Cleveland, Ohio.
Consulting Chemical Engineer, Industrial Testing Laboratory.

KINGSBURY, PERCY C., 50 Church St., New York, N. Y.
Chief Engineer German-American Stoneware Works, 50 Church St., New York City.

KIPPENBERG, HENRY, 15 Darmstadt Ave., Rahway, N. J.
Supt. of Chemical Manufacture at Rahway Plant of Merck & Co.

KREMER, WALDEMAR R., Vilter Mfg. Co., Milwaukee, Wis.
Electrical-Mechanical Engineer.

LAMAR, WILLIAM ROBINSON, 8–14 Johnson St., Newark, N. J.
President, Lamar Chemical Works.

LANGMUIR, ARTHUR C., 9 Van Brunt St., Brooklyn, N. Y.
Supt. Factory, Marx & Rawolle.

LARKIN, E. H., 3600 N. Broadway, St. Louis, Mo.
Director, National Ammonia Co., St. Louis, Mo.

LAZELL, E. W., 426 Railway Exchange Bldg., Portland, Ore.
Edwards & Lazell, Consulting and Chemical Engineers.

LEE, FITZHUGH, Grasselli Chemical Co., Cleveland, Ohio.
Assistant Chairman Manufacturing Committee, Grasselli Chemical Co.

LE MAISTRE, F. J., Ridley Park, Del. Co., Pa.
Chemical Engineer, E. I. du Pont de Nemours Powder Co.

LESSNER, C. B., Carril, Spain.
Manager of the Carril Works and Chemist to the San Finx Tin Mines, Ltd., and Metallurgical Chemist to the Angelita Mines.

LE SUEUR, ERNEST A., 50 McLaren St., Ottawa, Ont., Can.
General Manager and President of the General Explosives Co., Ltd.

LIHME, IENS P., Grasselli Chemical Co., Cleveland, Ohio.
Engineer, Grasselli Chemical Co.

LINDER, OSCAR, 56 North Waller Ave., Chicago, Ill.
Works Chemist, Western Electric Co., Hawthorne Works.

LITTLE, A. D., 93 Broad St., Boston, Mass.
President and General Manager, Arthur D. Little, Inc., Chemists and Engineers.
President and General Manager, Chemical Products Co., Boston, Mass.

LUNDTEIGEN, A., c/o Ash Grove Lime & Portland Cement Co., Kansas City, Mo.
Managing Engineer, Ash Grove Lime & Portland Cement Co., Kansas City, Mo.

MALLINCKRODT, EDWARD, St. Louis, Mo.
President Mallinckrodt Chemical Works.

MARSH, CLARENCE W., Niagara Falls, N. Y.
Chief Engineer The Development and Funding Co., New York City.

MASON, WILLIAM P., Troy, N. Y.
Prof. Chemistry, Rensselaer Polytechnic Institute.

MATOS, LOUIS J., 103 No. 19th St., E. Orange, N. J.
Technical Chemist and Chemical Engineer with the Cassella Color Co., 182 Front St., New York City.

MACNAUGHTON, WM. G., Port Edwards, Wis.
Assistant to General Manager in charge of manufacturing, Nekoosa Edwards Paper Co.

MCCORMACK, HARRY, Armour Institute, Chicago, Ill.
Professor of Chemical Engineering, Armour Institute of Technology, Chicago, Ill.; Editor of the Chemical Engineer; Consulting Chemist and Chemical Engineer.

MCKENNA, CHAS. F., 50 Church St., New York, N. Y.
Consulting Chemist and Chemical Engineer.

MEADE, RICHARD K., Roland Park, Baltimore Co., Md.
Consulting Chemical Engineer.

METZ, GUSTAVE P., 95 Elm St., Montclair, N. J.
Supt. and Vice-President, Consolidated Color and Chemical Co., Newark, N. J.

MILLER, A. L., 1104 Foulkrod St., Frankford, Philadelphia, Pa.
Supt. Chem. Dept., Barrett Mfg. Co.

MILLS, JAS. W., 2201 C St., Granite City, Ill.
Asst. Supt. Open Hearth Department Granite City Steel Works Branch of the National Enameling and Stamping Co.

MINER, H. S., Gloucester City, N. J.
Chief Chemist Welsbach Light Co.

MINOR, JOHN C., JR., 50 Church St., New York, N. Y.
Manager, General Carbonic Acid Co.

MYERS, RALPH E., 31 Franklin Ave., East Orange, N. J.

NEWHALL, CHAS. A., 603 Northern Bank Bldg., Seattle, Wash.
Senior Member of Newhall, Smith & Co., Chemical and Efficiency Engineers, Geologists.

OLNEY, LOUIS A., Lowell Textile School, Lowell, Mass.
Professor of Chemistry and Head of the Department of Textile Chemistry and Dyeing, Lowell Textile School; President Stirling Mills, Lowell, Mass.

OLSEN, JOHN C., Polytechnic Institute, Brooklyn, N. Y.
Prof. of Analytical Chemistry; Consulting Chemist.

PARKER, THOMAS J., 25 Broad St., New York City.
Chemical Expert of the Sales Department of the General Chemical Co.

PECKHAM, STEPHEN F., 150 Halsey St., Brooklyn, N. Y.

PORTER, J. EDWARD, Box 785, Syracuse, N. Y. Chemical Engineer.

PRENTISS, GEORGE N., 226 33d St., Milwaukee, Wis.
Chief Chemist, C., M. & St. P. R. R.

PUCKHABER, GEO. C., 805 Prospect Place, Brooklyn, N. Y.
Glue Maker and General Manager of Glue Department, Moller & Co., Maspeth, N. Y.

RICHARDS, J. W., University Park, South Bethlehem, Pa.
Professor of Metallurgy, Lehigh University; Secretary American Electrochemical Society; President Electrochemical Publishing Co.

REESE, CHARLES LEE, 725 du Pont Building, Wilmington, Del.
Chemical Director, High Explosives Operating Dept., E. I. du Pont de Nemours Powder Co.

ROBERTSON, ANDREW, 2 N. 9th St., Richmond, Va.
Member of firm Froehling & Robertson, Consulting Chemists and Chemical Engineers.

ROESSLER, FRANZ, 89 High St., Perth Amboy, N. J.
Vice-President and Secretary, Roessler & Hasslacher Chemical Co.

ROSENGARTEN, GEO. D., Box 1625; Philadelphia, Pa.
Vice-President The Powers-Weightman-Rosengarten Co.

PROCHAZKA, GEORGE A., 138 West 13th St., New York.
General Manager Central Dyestuff Chemical Co., Newark, N. J.

SADTLER, SAMUEL P., 39 South 10th St., Philadelphia, Pa.
Prof. Chemistry, Philadelphia College of Pharmacy, and Consulting Chemist (Samuel P. Sadtler & Son).

SADTLER, SAMUEL S., 39 South 10th St., Philadelphia, Pa.
Samuel P. Sadtler & Son.

SCHANCHE, H. G., 3500 Gray's Ferry Road, Philadelphia, Pa.
Chemical Director, Harrison Bros. & Co. Inc., Philadelphia, Pa.

SCHROEDER, C. M. EDW., Rutherford, N. J.
Consulting Chemist, 34 Bloomfield Ave., Passaic, N. J.

SHARPLES, STEPHEN P., 26 Broad St., Boston, Mass.
Analytical and Consulting Chemical Engineer and Assayer.

SHATTUCK, A. FORREST, Detroit, Mich.
Chief Chemist, Solvay Process Co.

SHIMER, PORTER W., Easton, Pa.
Proprietor and Chief Chemist of Chemical Laboratory.

SIMMONS, W. H., Fenton, Mich.
Superintendent, New Aetna Portland Cement Co.

SIMPSON, EDWARD H., Westside Ave., Jersey City, N. J.
Manager, Arlington plant of the Mutual Chemical Co. of America.

SMITH, ALBERT W., 7901 Euclid Ave., Cleveland, O.
Professor of Chemistry and Director of the Chemical Laboratory of Case School of Applied Science.

SMITH, FRANCIS PITT, 131–133 East 23d St., New York, N. Y.
Member of the firm of Dow & Smith, Chemical Engineers.

SMITH, HARRY E., 36 Beeresford Pl., East Cleveland, Ohio.
Chemist and Engineer of Tests, Lake Shore & Michigan Southern Ry.

SMITH, THEODORE E., 54 Hudson Place, Weehawken, N. J.
Supt. Oil Dept., Corn Products Refining Co., Edgewater, N. J.

SMITH, THORN, 125 Langley Ave., Detroit, Mich.
Diack & Smith, Consulting Chemists.

STILLMAN, JOHN MAXSON, Stanford Univ., Cal.
Professor of Chemistry.

TAKAMINE, JOKICHI, 520 West 173d St., New York City.
Consulting Chemist for Parke-Davis & Co., Detroit, Mich.

TAYLOR, EDWARD R., Penn Yan, N. Y. Manufacturing Chemist.

TAYLOR, JOHN, 137 S. New St., Bethlehem, Pa.

TEAS, WILLIAM H., Ridgway, Pa.
General Supt., U. S. Leather and Allied Companies.

THOMPSON, GUSTAVE W., 129 York St., Brooklyn, N. Y.
Chief Chemist, National Lead Co.

THOMSON, HENRY N., Tooele, Utah.
Superintendent and Consulting Metallurgical Engineer, Tooele Plant, International Smelting & Refining Co.

THORP, FRANK H., Boston, Mass.
Asst. Prof. Industrial Chemistry, Mass. Inst. Tech.

THIELE, LUDWIG A., Holland, Mich.
General Manager of the Holland Gelatine Works, Holland, Mich.

TOCH, MAXIMILIAN, 320 Fifth Ave., New York City.
Member of firm of Toch Bros.

TRAUTWEIN, A. P., Carbondale, Pa. Pres. Carbondale Instrument Co.

TUFTS, JOHN L., Winchester, Mass. Chemical Engineer.

TYSON, GEORGE N., 2815 Gray's Ferry Rd., Philadelphia, Pa.
Supt. for the Henry Bower Chemical Manufacturing Co.

VEILLON, A. A. L., 1800 South 2d St., St. Louis, Mo.
Vice-President and Works Manager, Monsanto Chemical Works.

VORCE, L. D., 364 W. Grand Boulevard, Detroit, Mich.
Supt. of Pennsylvania Salt Mfg. Co., Supt. of Goldschmidt Detinning Co., Gen. Supt. Wyandotte Southern Ry. Co.

WAGNER, THEODORE B., 17 Battery Place, New York, N. Y.
Operating Committee, Corn Products Refining Co.

WARREN, ROBT. C., 214 E. 5th St., Little Rock, Ark.
Chemical Engineer for the Arkansas Cotton Oil Co.

WATSON, JOHN R., P. O. Box 6, LaPlata, Md.

WESSON, DAVID, 111 South Mountain Ave., Montclair, N. J.
Manager Tech. Dept. Southern Cotton Oil Co., 24 Broad St., New York.

WHEELER, FRANK G., 778 Franklin St., Appleton, Wis.
Chemist with the Penn. Salt Mfg. Co., Wyandotte, Mich.

WHITAKER, M. C., Columbia Univ., New York City.
Professor of Engineering Chemistry, Columbia Univ. Editor of Jour. of Ind. and Eng. Chemistry, Consulting Chemical Engineer.

WHITE, FRED. S., 392 Clinton Ave., Brooklyn, N. Y.
Supt. Glycerine Dept., Marx & Rawolle, Brooklyn, N. Y.

WIECHMANN, FERDINAND G., 39 W. 38th St., New York, N. Y.
Consulting Chemical Engineer.

WILLIAMS, FRANK M., 43–44 Sherman Bldg., Watertown, N. Y.
Consulting Chemical Engineer and Industrial Chemist.

WITHROW, JAS. R., Columbus, O.
Professor of Chemistry, Ohio State University, Columbus, O.

WOOD, F. J., 361 Henry St., Brooklyn, N. Y.
Chief Engineer, Marx & Rawolle.

WURSTER, O. H., Toronto, Canada.
Assistant Works Manager, Lever Bros., Ltd., Toronto.

ZINSSER, FREDERICK G., Hastings-on-Hudson, N. Y.
Manufacturing Chemist under firm of Zinsser & Co.

ZITKOWSKI, HERMAN E., Rocky Ford, Colo.
Chief Chemist and Technical Adviser, American Beet Sugar Co., Denver, Colo.

ZWINGENBERGER, O. K., P. O. Box 112, Perth Amboy, N. J.
Patent Dept., Roessler & Hasslacher Chem. Co.

JUNIORS

ALLEN, WILLIAM P., 83 Aberdeen Place, Woodbury, N. J.
Chemist, E. I. du Pont de Nemours Powder Co.

BICKNELL, R. S., 50 E. 41st St., New York, N. Y.
Chemical Engineer, The Thermal Syndicate, Ltd., New York City, N. Y.

BOYLSTON, ARTHUR C., 3600 N. 2d St., St. Louis, Mo.
Chemist in charge of Manufacturing with the Mallinckrodt Chemical Works.

BUCKMAN, HENRY H., Indianapolis, Ind.
Chief Chemist, American Hominy Co.

CAMPBELL, CHARLES L., Wallaston, Norfolk Co., Mass.
Chemical Engineer, with E. B. Badger & Sons Co., 75 Pitts St., Boston, Mass.

CLARK, WM., Crafton, Pa.
Metallurgist and Chief Chemist, American Vanadium Co.

FALES, H. A., 308 Schermerhorn St., Brooklyn, N. Y.
Vice-President and Secretary of The W. H. Fales Co.

GAGE, R. M., The Oaks, Springfield, Mass.
Chemical Engineer, The Fisk Rubber Co., Chicopee Falls, Mass.

GUILLAUDEU, ARTHUR, Cincinnati, O.
Chemist and Asst. Supt., The M. Werk Co., 408–432 Poplar St., Cincinnati, O.

HEINRICH, E. O., 3214 North 30th St., Tacoma, Wash.
Consulting and Manufacturing Chemist; Director of the Heinrich Technical Laboratories.

JORDAN, HARRY E., 113 Monument Place, Indianapolis, Ind.
Sanitary Engineer, Indianapolis Water Co.

LANE, FRED. H., Hollis, L. I., N. Y.
Chemical Superintendent, Emil Calman & Co., Vernon and Harrison Aves., Long Island City, N. Y.

LAWRENCE, JAS. C., P. O. Box 812, Memphis, Tenn.
Consulting Chemical Engineer, Directing Engineer, Forest Product. Chemical Co., Memphis, Tenn.

LUNN, CHARLES ALBERT, Brunswick, Ga.
Chemical Engineer, The Yaryan Naval Stores Co.. Brunswick, Gas

LUNT, G. P., 75 Pitts St., Boston, Mass.
Chemical Engineer, E. B. Badger & Sons Co.

MCINTYRE, A. G., 404 McGill Bldg., Montreal, Canada.
Chief, Forest Products Laboratories of Canada. Editor, Pulp and Paper Magazine of Canada.

MEADE, GEORGE P., Gramercy, La.
Asst. Supt. and Chief Chemist, Gramercy Refinery, Colonial Sugar Co.

MITKE, CHARLES A., Box 1226, Dawson, N. M.

PETERSON, CHARLES ALBERT, Lancaster, Pa.
Chief Chemist in the linoleum dept. of the Armstrong Cork Co., Lancaster, Pa.

PLUMB, ROY A., 58 Lafayette Ave., Detroit, Mich.
Director of Michigan Technical Laboratory, 58 Lafayette Boul., Detroit, Mich.

SCHAEFFER, JOHN A., Joplin, Mo.
Chief Chemist, Picher Lead Co., Joplin, Mo.

SEAMAN, E. H., Wantagh, L. I.
Insurance Engineer, with Underwriters at American Lloyds.

SHREVE, R. NORRIS, care Lamar Chemical Works, Newark, N. J.

SUMMERS, FRANK P., 1525 Winnemac Ave., Chicago, Ill.
Chemist in charge, Abbott Alkaloidal Co., Chicago, Ill.

TURNER, NORMAN L., Belleville, Ontario, Can.
Provincial Assayer of Ontario, Can.

VAN DOREN, WILLARD V., 1646 Garfield Boulevard, Chicago, Ill.
Superintending Chemist of the plant of the Illinois Vinegar Mfg. Co., 48th St. & Oakley Ave., Chicago, Ill.

WHITCOMB, L. R., 100 William St., New York.
Chemist and Bacteriologist in charge of Testing Lab., for Nicholas S. Hill, Jr.

INDEX

PAGE

Acetylene, solubility of 137
solvents for 133
Acetaldehyde as solvent for acetylene 140, 143, 144, 145
Acetone as solvent for acetylene 141
Action of disinfectants on sugar solutions 88
Asphaltic rocks of the United States and their use in street paving 245
Asphalts and petroleums, the presence of oxygen in 178
Availability of blast-furnace slag as a material for building brick 204

Baekeland, L. H., Phenol-Formaldehyde Condensation Products 1
Protection of Intellectual Property in Relation to Chemical Industry 19
Bagasse 229
Bakelite 1
Beehive coke oven industry of the United States 78
Beet sugar, process of manufacture 222
Belden, A. W., the beehive coke oven industry of the Unted States 78
Blast-furnace slag, availability of as a material for building brick 204
Booth, Wm. M., the chemical engineer and industrial efficiency 184
water for industrial purposes 197
Building brick, Availability of blast-furnace slag as a material for 204
tests for 208

Campbell, John Hayes, The need of standard specifications in oils for paving block impregnation 170
Cane sugar, process of manufacture of 225
Chemical control and technical accounting in sugar manufacture 220
Chemical engineer and industrial efficiency 184
Chemical engineering course and laboratories at Columbia University. 150
Chemical investigation of Asiatic rice 70
Code of Ethics 255
Coggeshall, Geo. W., and A. S. Cushman, Production of available potash from the natural silicates 52

PAGE

Columbia University, New chemical engineering course and laboratories at 150
Column still and extractor 158
Committees for 1913 267
Constitution 259
Control of initial setting time of Portland cement 119
Creosote oil, Analyses of 174
Cushman, A. S., Study of the temperature gradients of setting Portland cement 43
and Geo. W. Coggeshall, production of available potash from the natural silicates 52
and H. C. Fuller, Chemical investigation of Asiatic rice 70

Davoll, Jr., David S., Technical accounting and chemical control in sugar manufacture 220
Decomposition of linseed oil during drying 100
Disinfectants, action of, on sugar solutions 88
Drinking water 200

Effect of "lime sulphur" spray manufacture on the eyesight 127
Ester-ketone-aldehyde as solvent for acetylene 142
Ethics, Code of 255

Feldspar, potash, silica, and alumina from 68
Formaldehyde, action of phenol on 8
Fuller, H. C., and A. S. Cushman, a chemical investigation of Asiatic rice 70

Hart, Edward, potash, silica, and alumina from feldspar 68

Industrial efficiency and the chemical engineer 184
Industrial purposes, water for 197
Intellectual property, protection of in relation to chemical industry 19

James, J. H., acetylene solvents 133

"Lime sulphur" spray manufacture, effect of on the eyesight 127
Linseed oil, decomposition of, during drying 100

Manufacture, statistics of 192
Massecuite 235
Meade, Geo. P., Action of disinfectants on sugar solutions 88
Members for 1913 269

PAGE
Notes on a study of the temperature gradients of setting Portland cement. 43

Officers for 1913 266
Olsen, J. C., and A. E. Ratner, decomposition of linseed oil during drying 100
Opacimeter, Thompson's 111
Opacity and hiding power of pigments, tests on 108
Oxygen in petroleums and asphalts 178

Paper laboratory 160
Patent, question, discussion of 37
system of the United States 21
Paving block impregnation, need of standard specifications for oils in . . . 170
Peckham, S. F., the asphaltic rocks of the United States and their use in street paving 245
Petroleums and asphalts, the presence of oxygen in 178
Phenol-Formaldehyde condensation products 1
Pigments, tests on the opacity and hiding power of 108
Portland cement, control of initial setting time of 119
Notes on a study of the temperature gradients of the setting of 43
Potash, from natural silicates, processes proposed for 57
production of, from the natural silicates 52
silica and alumina from feldspar 68
Production of available potash from the natural silicates 52
Protection of intellectual property in relation to chemical industry 19

Ratner, A. E., and J. C. Olsen, decomposition of linseed oil during drying 100
Rice, Chemical investigation of 70
Composition of 72, 76

Sadtler, Sam. P., The presence of oxygen in petroleums and asphalts . . . 178
Shelf drier and vacuum pumps 159
Silicates, production of potash from 52
Standard specifications in oils for paving block impregnation 170
Sugar, analysis of raw material for 228
manufacture, technical accounting and chemical control in 220
solutions, action of disinfectants on 88
table of purities for 244
Table of Purities for sugar 244
Technical accounting and chemical control in sugar manufacture 220
Temperature gradients of setting Portland cement 43
Tests on the opacity and hiding power of pigments 108

PAGE

Thompson, G. W., Tests on the opacity and hiding power of pigments. 108
Thompson's opacimeter 111

Vacuum pans 157

Ware, E. E., control of initial setting time of Portland cement 119
Water for industrial purposes 197
Whitaker, M. C., New chemical engineering course and laboratories at Columbia University 150
White, Albert E., Availability of blast-furnace slag as a material for building brick 204
Withrow, James R., The effect of "lime sulphur" spray manufacture on the eyesight 127

PERIODICAL